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# Investigation of Textile Dyebath Reconstitution and Reuse. Volume 1 Technical Report

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# INVESTIGATION OF TEXTILE DYEBATH RECONSTITUTION AND REUSE

## Volume 1. Technical Report

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by

Jon F. Bergenthal

Anthony J. Tawa

SVERDRUP & PARCEL AND ASSOCIATES, Inc.
St. Louis, Missouri 63101

EPA Project Officer: Robert V. Hendriks

Industrial Environmental Research Laboratory
Utilities and Industrial Processes Division
Research Triangle Park, NC 27711

INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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| Textile Finishing | Stationary Sources | 13H |
| Dyeing | Dyebaths | |
| Water Treatment | Reconstitution and Reuse | |
| Waste Water | Carpet Mills | |

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# ABSTRACT

Approximately 80 percent of textile finishing mills discharge their wastewater to publicly owned treatment works. The majority of the wastewater receives little or no pretreatment before discharge into municipal collection systems. A variety of wastewater recycle/reuse technologies allowing these mills to reduce the volume of wastewater and the amount of pollutants discharged were described in the report covering Phases I and II of this project.

This report, covering Phase III of the project, examines one of these recycle technologies, dyebath reconstitution and reuse, in detail. This recycle technology is considered promising for a number of reasons including its significant environmental benefits, potential for widespread application, low capital cost, cost savings in textile dyeing, and economic attractiveness.

Volume 1 of this report presents the results of a detailed investigation of dyebath reconstitution and reuse at a carpet mill. The results of bench-, pilot-, and full-scale testing are presented. Wastewater data documenting the pollutant reductions achieved through dyebath reuse are presented. The economic feasibility of implementing the technology full-scale is addressed.

Volume 2 of this report presents detailed procedures and methods for implementing dyebath reconstitution and reuse. Its purpose is to serve as an operations manual for other mills wishing to investigate this recycle technology.

This report is submitted in accordance with Contract Number 68-02-3678 between the U.S. Environmental Protection Agency and Sverdrup & Parcel and Associates, Inc. It documents the work performed under Phase III of the contract.

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# UNIT CONVERSION TABLE

### Flow/Volume

1 gallon = 0.003785 $m^3$
1 gpm (gallon per minute) = $6.308 \times 10^{-5} m^3/s$
1 mgd ($10^6$ gallon/day) = 0.0438 $m^3/s$

### Mass

1 grain = $6.48 \times 10^{-5}$ kg
1 ounce = 0.0284 kg
1 lb = 0.4536 kg
1 ton = 907.2 kg

### Length

1 inch = 0.0254 m
1 foot = 0.3048 m
1 yard = 0.9144 m

### Time

1 minute = 60 s

### Energy, Power

1 Btu = 1.055 kJ
1 HP (horsepower) = 0.7457 kW

### Temperature

$°F = °C \times 9/5 + 32$
$°C = [°F - 32] \times 5/9$

### Miscellaneous

1 gal/lb = 0.00834 $m^3/kg$

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# SECTION I
# INTRODUCTION

Over four million tons (3.6 x $10^9$ kg)* of textile products are finished annually in the United States, resulting in the discharge of more than 100 billion gallons of wastewater (1, 2). It is estimated that about 80 percent of textile finishing mills discharge their wastewater to publicly owned treatment works (POTW) (2). Most of this wastewater receives either no treatment or only preliminary treatment, such as settling, screening, equalization, or neutralization, prior to discharge to the municipal sewers.

The major textile finishing operations include fiber preparation (desizing, scouring, mercerizing, bleaching), fiber coloring (dyeing, printing), and functional finishing. Wastewater results primarily from the preparation and coloring operations. Preparation wastewaters contain impurities removed from the fibers during desizing and scouring and also contain excess, unused processing chemicals such as desizing agents, scouring agents, caustic, and bleaches. Coloring wastewaters contain unexhausted dyes or printing pigments along with auxiliary processing chemicals. Many wastewater recycle/reuse technologies are available to allow reuse of these wastewaters. These technologies are described in detail in the report covering Phases I and II of this project. One of these technologies, dyebath reconstitution and reuse, appeared to offer significant environmental benefits and substantial cost savings to the industry, and it also has the potential for widespread use within the industry. This report presents the results of a detailed investigation of dyebath reconstitution and reuse.

The vast majority of textile products are colored by dyeing. In 1980, approximately 7.8 billion pounds (3.9 million tons) of fibers were dyed. To accomplish this dyeing, 171 million pounds of dyestuffs and 1.28 billion pounds of additives (auxiliary chemicals) were used (3). Most auxiliary chemicals are not taken up by the fibers

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* The units of measurement most familiar to the projected readership of this report have been maintained.

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(exhausted) during dyeing and are thus discharged with the dyeing wastewater. Dyestuffs, however, generally exhaust to over 90 percent during dyeing. Ninety-five percent is an often quoted value for the average dyestuff exhaustion during dyeing. Assuming a 95 percent exhaustion rate for the dyestuffs still leaves about 8.5 million pounds of dyestuffs discharged annually in dyeing wastewater.

Dyeing can be performed using either continuous or batch operations. Continuous dyeing is presently limited to the broadwoven and carpet segments of the industry where high volume, long color runs are more common. Recent data indicate that about 50 percent of all textile dyeing on a weight basis is performed on continuous ranges. The balance is performed using batch dyeing. Batch dyeing remains popular due to its flexibility, short-run capability, and ease of control. Batch processes, however, are generally inefficient in their usage of water and auxiliary chemicals.

In a typical batch dyeing operation, from one to five gallons of water are used as dye liquor for each pound of fiber dyed. Auxiliary chemicals and dyes are added to this dye liquor. Auxiliary chemicals can include exhaust agents, leveling agents, buffers and pH control chemicals, retarding agents, wetting and dispersing agents, carriers, softeners, lubricants, penetrants, among many others. The total amount of auxiliary chemicals added will vary depending on the fiber and dyestuff types, but will generally range from a few percent of the fiber weight (2 or 3 percent o.w.f.) to as much as 50 percent o.w.f. where high concentrations of exhaust agents are required. Dyestuff quantities are generally a few (less than 4) percent of the fiber weight. Following the addition of auxiliary chemicals and dyes, the dyebath temperature is raised to the desired dyeing temperature and held until dyeing is complete and a level dyeing is achieved. The exhausted dyebath, now containing only a few percent of the original quantity of dyestuff but still most of the auxiliary chemicals, is dropped, and the dyed product is rinsed with fresh water.

The possibility exists of reusing the exhausted dyebath for subsequent dyeings, thus making use of the auxiliary chemicals for more than one cycle of dyeing. This results in production cost savings and also decreases the volume of wastewater and quantity of pollutants discharged. To realize this possibility of dyebath reuse, two things had to be developed:

1. A method for analyzing the exhausted dyebath to determine the quantities of dyestuffs remaining and thereby compute the add quantities for the next dyeing.
2. A demonstration that dyeing could be successfully performed with recycled dyebaths, producing acceptable shade matching, levelness, and product quality.

Most of the work in the 1970's developing the dyebath analytical techniques and reconstitution methods was performed at the School of Textile Engineering at the Georgia Institute of Technology. The success of these bench-scale and pilot-scale tests led to three full-scale demonstrations, also by GIT, the results of which are detailed in references 4, 5, and 6, listed in this report.

The work conducted under Phase III of the EPA Textile Wastewater Recycle/Reuse project expands upon the work conducted previously on dyebath reconstitution and reuse in the following ways:

1. It examines the application of dyebath reuse at a mill that is fairly typical of many dyehouses, yet is not a "perfect" candidate for this technology.
   - Dye formulations had to be modified to use a smaller number of dyestuffs before reconstitution became technically feasible.
   - Quality control requirements are strict at this mill, thus posing a stiff test of the ability of dyebath reuse to produce acceptable dyeings.
   - Overflow rinsing is typically used at this mill, thereby affecting not only the dyebath analysis/reconstitution methods, but also the economics of dyebath reuse.
2. It presents a very detailed examination of the techniques used not only in full-scale testing but also in the development and testing of the technology at this mill. These techniques can be directly applied by other mills wishing to examine the feasibility of dyebath reuse.

3. It presents the first development of environmental data for this wastewater recycle/reuse technology. Thus, the environmental benefits of dyebath reuse can be quantified for the first time.

4. It updates the equipment used in dyebath analysis to include a modern desk-top computer, a commercially available interface for the spectrophotometer/computer, and software written in the BASIC computer language to allow for greater comprehension and adaptability to a variety of desk-top computers.

It was determined that the best way to develop the above information was to examine dyebath reconstitution in detail at a textile mill. A carpet mill, Mill C-2 which participated in the Phase II studies, was selected for the full-scale evaluation. Mill C-2 primarily performs atmospheric batch dyeing of nylon carpet, discharging about 1 mgd of wastewater to the municipal collection system. The project consisted of the following steps:

- Reformulate dye recipes to use fewer dyestuffs and allow dyebath reconstitution.
- Select product styles and shades appropriate for dyebath reuse testing.
- Collect data on dyestuffs necessary for dyebath analysis.
- Test dyebath analysis/reconstitution techniques on a bench-scale using beaker dyeing to refine the dyebath analysis techniques and check shade matching.
- Develop hardware and software needed for quick and easy analysis and reconstitution of dyebaths on a plant scale.
- Test dyebath reconstitution and reuse on a pilot-scale to enable product quality evaluation.
- Test dyebath reconstitution and reuse in a full-scale beck to evaluate dyebath reuse feasibility.
- Collect dyebath samples to measure the environmental benefits of dyebath reuse.
- Perform an economic analysis of dyebath reuse.
- Prepare a report that details the work performed and serves as a guide for other mills wishing to investigate dyebath reuse.

This report presents the results of the above work. It is organized as follows: Section II presents the conclusions and recommendations of the study. Section III presents a general discussion of the concepts behind dyebath reconstitution and the selection of dyestuffs, shades, and products for use in dyeing using reconstitution techniques. The reformulation of dye recipes to utilize a smaller number of dyestuffs is also discussed.

Section IV explains the theory behind the dyebath analysis and reconstitution techniques and describes the necessary equipment and procedures.

Section V presents a description of the computer and programs used in the pilot-scale and full-scale tests to simplify the dyebath analytical and reconstitution calculations.

Sections VI, VII, and VIII present the results of bench-scale, pilot-scale, and full-scale testing, respectively. Section IX describes the environmental data collected and quantifies the environmental benefits of dyebath reuse. Section X presents an economic analysis of dyebath reuse for this case.

Volume 2 of this report is an operations manual providing a complete and easily read guide to dyebath reuse technology. This guide is compatible with conventional dyehouse operation and should provide sufficient information to permit mills to evaluate this wastewater recycle/reuse technology.

# SECTION II

# CONCLUSIONS AND RECOMMENDATIONS

## CONCLUSIONS

This project demonstrated the technical feasibility of batch dyebath reconstitution and reuse at a carpet mill. It demonstrated how several technical problems common to a wide variety of mills were overcome. These problems included selecting product styles and shades that can be incorporated in a dyebath reuse scheme; reformulating dye recipes to use a single group of dyestuffs to dye many shades; demonstrating that dyebath reuse can be accomplished on a portion of production within the normal production schedule; producing high-quality product with recycled dyebaths; and adapting the dyebath reuse procedures to conform to the mill's standard dyeing procedures, including such considerations as initial dyebath temperature and the use of overflow cooling.

Environmental benefits are significant, even when dyebath reuse is practiced in a non-optimal fashion. Pollutant and water use reductions of 25 to 50 percent were observed. Environmental benefits are maximized when dilution of the dyebath through steam condensation and overflow cooling is kept to a minimum.

Dyebath reuse is economically attractive, even when practiced in a non-optimal fashion. A payback period of 1.5 years was determined for Mill C-2 in this study. Again, economic benefits are maximized when dilution of the dyebath is minimized.

Because of the large amount of batch dyeing performed in the industry, and the low capital cost and attractive economics of dyebath reuse, this wastewater recycle/reuse technology can be expected to grow in use. Specific areas for use include knit fabric dyers, carpet dyers, and yarn dyers. Larger hosiery mills may also find dyebath reuse advantageous.

One major obstacle to the further use of this technology is the lack of detailed information in the literature on the equipment, analytical techniques, and procedures for dyebath reuse.

RECOMMENDATIONS

1. The information in the two volumes of this report should be disseminated to encourage the use of dyebath reconstitution/reuse.

2. If dyebath reuse is instituted at Mill C-2, the dyebath reuse procedures should be optimized to minimize cooldown rinse water requirements. This will improve the economic attractiveness of dyebath reuse as well as further reduce the discharge of wastewater.

# SECTION III

# DYEBATH RECONSTITUTION CONSIDERATIONS

Dyebath reconstitution is an attractive wastewater recycle/reuse technology in certain situations; however, it is not applicable to all dyeing operations. This section briefly reviews the concept of dyebath reconstitution. This is followed by a discussion of applicable reuse situations as well as limitations. This section concludes with a description of dye recipe reformulation.

## CONCEPT

The two basic methods used for textile dyeing are continuous processing and batch processing. Batch processes are much less efficient than continuous processes in their use of chemicals, water, and energy, but are often more attractive to textile mills due to their versatility, ease of control, and short run capability. The following observation illustrates why reconstitution and reuse of batch dyebaths is a potentially worthwhile water reuse technology: "Comparison of the dyebath at the beginning and at the end of the dyeing reveals that the major change that occurs during the dyeing is the transfer of dye from the bath to the (product) and an increase in temperature from ambient to (elevated). The dyeing assistants, pH control agents, and the water are essentially unchanged by the dyeing process. It should be possible therefore to analyze the bath following the dyeing process and to determine the quantity of dye that must be added to reconstitute the bath to the desired concentration for a subsequent dyeing" (7). Simply stated, dyebath reconstitution is the adding of appropriate quantities of make-up dyes, and auxiliary chemicals as applicable, to an exhausted dyebath to enable subsequent reuse for additional dyeings.

Since dyebath reconstitution is not a capital-intensive reuse technology, and it can provide significant chemical, energy, and water savings, the economics are usually quite favorable with a relatively short payback period. Therefore, the key to the feasibility of dyebath reconstitution at a given mill is more likely to depend on technical considerations than economic considerations. And although reconstitution is a simple, straightforward concept, it is not applicable to all batch dyeing situations.

## APPLICATIONS

As noted above, dyebath reconstitution is applicable primarily to batch dyeing processes. In continuous dyeing, where ultrafiltration or hyperfiltration can be used to recover a concentrated dyestuff stream from rinse water, dyebath reconstitution techniques may also be applied to allow concentrate stream reuse. However, this application is still under development, particularly at Riegel Textiles (LaFrance Industries). This report deals only with applications to batch dyeing. Textile products that are generally dyed by batch operations include hosiery and other knit goods, yarn, beck-dyed carpets, and some woven goods.

Several types of dyes, classified on the basis of their dyeing properties, are currently used in the textile industry. These include disperse, acid, basic (cationic), direct, reactive, sulfur, and vat dyes. A breakdown of the primary fibers and dyes for products most commonly dyed by batch processes is listed below:

| Product | Primary Fibers | Dyes |
|---|---|---|
| Ladies' hosiery | nylon | acid, disperse |
| Mens' hosiery | nylon, acrylic, cotton | acid, basic, direct |
| Knit fabric | polyester, cotton, nylon, acrylic | disperse, direct, reactive, acid, basic |
| Yarn | cotton, polyester, acrylic, nylon | direct, reactive, vat, disperse, basic, acid |
| Carpet | nylon, polyester | acid, disperse |
| Woven fabric | polyester, cotton | disperse, direct reactive, vat |

From this table it can be seen that acid and disperse dyes (used for nylon and polyester fibers) are widely used in batch dyeing. It is not surprising, then, that most of the dyebath reconstitution efforts to date have focused on acid and disperse dyes. Based on experiments and successful reconstitution experiences, this reuse technology is known to be applicable to these two dyestuff classes. A successful application for batch dyeing of acrylic fibers with basic dyes is also known. For cotton fibers, reconstitution and reuse has been investigated for direct and reactive dyes. A special split-bath technique is required with reactive dyes. Little research and/or information is available regarding the applicability of reconstitution to the lesser used dye classes such as azoic, mordant, premetallized, sulphur, and vat dyes.

A series of dyeings with dyebath reconstitution and reuse must use the same dyestuffs in each dyeing (although in different proportions). Therefore, for reconstitution to be feasible, a mill must perform a certain amount of similar dyeing. For example, reconstitution may not be appropriate for certain commission finishers where product, dyes, chemicals, and lot size are constantly changing. However, where the same few combinations of dyes are used for a regular portion of a mill's output, reconstitution may be applicable. And even if a mill presently uses a large variety of dyestuffs, reformulation of dye recipes to reduce the number of dyestuffs used for a significant portion of production can clear the way for reconstitution to be implemented. This is discussed below.

A further consideration is that even where technically feasible, dyebath reconstitution and reuse will generally be instituted on only a portion of a mill's dyebecks. One half of the dye machines is a commonly used goal. This is to ensure that flexibility in production is maintained.

## REFORMULATION OF DYE RECIPES

Until recently, Mill C-2 used a large number of dyestuffs in a plethora of combinations to produce the numerous shades in their product lines. However, partly to facilitate dyebath reuse, but also to achieve economies of scale through bulk purchases and simplified inventories, the mill has reformulated most of their dye recipes. At present, only a half dozen or so dyes, divided among the three colors (red, orange/yellow, and blue) and formulated into approximately the same number of three-dye combinations, are necessary for the majority of the dyeings at the mill.

The principal dyes used in the reformulated recipes were selected by experienced mill personnel based on the dyeing characteristics and economics of the individual dyes. Computer shade matching using a Diano/Hardy II spectrophotometer in conjunction with laboratory beaker dyeings were used to quickly reformulate old recipes for given shades to use these principal dyes. This reformulation of dye recipes removed a major constraint to dyebath reconstitution and reuse at this mill.

## ADDITIONAL INFORMATION

This report examines investigations at one mill only; as noted in the introduction, it expands upon and supplements other research. In particular, the reader interested in additional information on reconstitution investigations at hosiery mills (Ref. 4, 8, 9 and 10), yarn mills (Ref. 1, 8 and 9), and jet dyeing mills (Ref. 5) should review the referenced reports. An excellent discussion of shade sequencing and optimum length for reconstitution series is presented in Reference 11.

Volume 2 of this report provides more detailed information on the procedures for and implementation of dyebath reuse.

# SECTION IV
# DYEBATH ANALYSIS

The underlying principle enabling dyebath analysis is the fact that most dyes exhibit strong absorption of light in the visible region of the spectrum. Each dyestuff has a characteristic absorbance of light at various wavelengths of the spectrum. This absorbance is proportional to the concentration of the dyestuff in the dyebath and can be measured using a spectrophotometer. As a result, the residual concentrations of each dyestuff in an exhausted bath can be determined. This enables calculation of the quantities of dyestuffs that must be added to reuse the dyebath for the next dyeing.

In this section, the principles of absorbance and its measurement are discussed first, together with the application of these principles to dyebath analysis. The analytical procedures used for dyebath analysis are then presented. Preparatory activities required to establish the absorbance characteristics of dyestuffs are discussed next, and the data developed for the dyes used in this demonstration study are presented. Finally, the use of absorbance data to determine residual dye concentrations and make-up amounts for dyebath reuse is illustrated.

## DYEBATH ANALYSIS PRINCIPLES

### Absorbance and Its Measurement

A detailed discussion of colorimetry and absorbance can be found in references 12 and 13 listed in this report. Only a brief summary of the subject, as it directly applies to dyebath analysis, is presented here.

Radiant energy in a beam of light passing through an exhausted dyebath will emerge less powerful than it entered, due to absorption of some of the light by the dyebath. The behavior of light transmitted through this exhausted dyebath can be modeled by combining Lambert's law (equal thicknesses of an absorbing solution absorb an equal fraction of the light tranversing it) and Beer's law (the intensity of transmitted light at a given wavelength decreases exponentially as the concentration of the absorbing medium increases) to give the following Lambert-Beer equation:

$$A = \log I_o/I = kC$$

where $A$ = absorbance of the dye
$I_o$ = the intensity of light entering the exhausted dyebath
$I$ = the intensity of light leaving the exhausted dyebath
$k$ = constant for the particular dye at a given wavelength
$C$ = the dye concentration in the exhausted dyebath

Also, because absorption is an additive property, the absorbance of the light at a given wavelength in a mixture of dyes is the sum of the absorbances of each of the dyes at that wavelength, i.e., $A = A_1 + A_2 + A_3 + \ldots = k_1C_1 + k_2C_2 + k_3C_3 + \ldots$

Absorbance is commonly measured using a spectrophotometer, an instrument ideally suited for this task due to its ability to produce and measure monochromatic light* at any wavelength selected by the operator. This monochromatic light is produced by passing ordinary light through a dispersing element (a diffraction/reflection grating or a prism) in combination with very thin entrance and exit slits to isolate and direct an extremely narrow band of wavelengths. The resulting monochromatic light passes through a cuvette containing an exhausted dyebath sample and emerges with a decreased intensity due to absorbance by the sample. A solid state or photoelectric tube detector is located in the path of this emergent light, and the current developed by this detector is translated into an absorbance reading by electronic circuitry.

### Applicability of Absorbance to Dyebath Analysis

As seen above, the spectrophotometer can be used to determine the absorbance of an exhausted dyebath sample. When only a single dyestuff is present in a dyebath, its concentration is related to this absorbance by $A = kC$. However, since nearly all dyebaths are solutions of two or more dyestuffs that exhaust unequally, the additive characteristic of light absorption by dyes becomes important to dyebath analysis. To analyze for several dyes in a bath, the absorbance is measured at a number of wavelengths (usually corresponding to the number of dyes present). The concentrations of the dyes are then determined by simultaneous solution of a set of Lambert-Beer linear equations of the form $A = k_1C_1 + k_2C_2 + k_3C_3 + \ldots$. One equation results from each analysis wavelength.

---

*Monochromatic light consists of radiation of a single wavelength or a small range of wavelengths; each wavelength in the visible spectrum is associated with a specific color or hue.

Selection of the wavelengths at which absorbance measurements are made, and determination of the dye absorbance constants (k-values) are necessary prerequisites to this activity. The remainder of this section presents the analytical procedures used to obtain absorbance measurements, illustrates the determination of wavelengths and k-values and shows how residual dye concentrations are calculated and used to determine dyestuff requirements for subsequent dyeings.

## ANALYTICAL PROCEDURES

### Sample Preparation

Exhausted dyebaths contain, in addition to unexhausted dyestuffs, various "contaminants" such as fiber particles, auxiliary chemicals, fiber finishes, greige goods tints, etc. These additional substances can absorb or scatter light thus interfering with accurate absorbance readings of residual dyestuffs. Therefore, procedures for their removal from samples to be spectrophotometrically analyzed may be required. Sample preparation was needed for accurate analysis of dyebaths during this study, and two approaches, filtration and solvent extraction, were examined.

Filtration. This method of sample preparation removes fibers and other filtrable solids by forcing the exhausted dyebath through a filter under pressure. It is thus appropriate for water soluble dyestuffs in dyebaths where no soluble interferences are present. The following equipment was used in the filtration procedure: 1) 20-cc glass Luer-Lok tip syringe; 2) Swinny filter attachment with teflon O-ring, stainless steel filter screen, and teflon gasket; 3) Swinny water filters; and 4) prefilters. The syringe is prepared each time by placing a clean water filter and prefilter within the Swinny filter attachment. Exhausted dyebath sample is added into the barrel of the syringe and is forced through the filters into a receiving cuvette by applying pressure to the plunger. This procedure is effective in removing suspended solids from the exhausted dyebath, but does not remove water soluble tints or other dissolved solids that can cause interference. If such interferences are present in the sample, solvent extraction is an alternative to filtration for sample preparation.

Solvent Extraction. This method takes advantage of the fact that certain dye classes are extractable into organic solvents. Disperse dyes, for example, are not water soluble and must always be extracted in an organic solvent such as benzene or toluene. Many dye classes such as acid and basic dyes will dissolve in alcohols. Based on research by the Georgia Institute of Technology at Salem Carpet Company, (6), it is known that acid dyes are extractable into octanol, whereas greige good tints are not. Therefore, this procedure was ideally suited for this study.

The following equipment was used to perform the solvent extraction procedure in this study: 1) 1-Octanol, certified grade; 2) table salt; 3) cotton balls; 4) two 25-ml graduated cylinders, 5) 250-ml separatory funnel; 6) 20-cc glass Luer-Lok tip syringe; 7) 20-ml sample vials; and 8) ring stand and attachments. Twenty ml (approximately 25 grams) of salt, 25 ml of exhausted dyebath sample, and 25 ml of octanol are added in succession to the separatory funnel. The funnel is stoppered and shaken vigorously for 30 seconds. The water and octanol layers are allowed to partially separate in the funnel before again being shaken for approximately 30 seconds. Next, the funnel is placed on the ring stand to allow distinct separation of salt (bottom of funnel); water, i.e., exhausted dyebath minus dye (middle layer); and octanol with extracted dyes (top layer). The stopcock is opened, the salt and water layers are allowed to drain out, and the stopcock is closed. The octanol layer is then drained into the syringe into which two cotton balls have been placed to absorb any water present in the sample. Finally, the extracted sample is collected in a vial as it passes through the cottonballs and out of the syringe.

For comparative purposes, both sample preparation approaches were compared during k-value determinations and beaker dyeing trials. Based on this comparison, solvent extraction was determined to be more reliable than filtration and was employed in subsequent phases of the project. An added advantage of this method is that samples and standards prepared in this manner are not subject to biological degradation and can therefore be stored for extended periods, if desired.

### Absorbance Measurements

To be meaningful, absorbance readings using a spectrophometer must be made against a reference standard or blank to correct for absorbance or scattering of light by species other than dyes in the sample. In analyzing exhausted dyebaths, the most appropriate blank is one that is identical to the exhausted dyebath in all respects with the exception that no dyestuffs are present. In this study, these blanks were prepared by performing a simulated bench-scale "dyeing" of the carpet with appropriate liquor ratios, auxiliary chemicals, and temperatures, but without dyestuffs. An extracted sample of this mock dyebath was then used as the reference blank in the spectrophotometer.

A Bausch & Lomb Spectronic 21-DV digital display spectrophotometer and its associated general-purpose sample compartment were used in this project. The Spectronic 21-DV is a single-beam, grating spectrophotometer with a spectral range from 340 to 1,000 nm. The attached sample compartment allows up to three cuvettes to be placed in the multiple sample holder; the appropriate cuvette is moved into the light path for an absorbance reading by operating a push-pull lever. The cuvettes used were 10-mm rectangular spectrophotometer cells with standard silica windows.

The procedure used during this project in making absorbance readings at a given wavelength was to place the reference blank in the first sample holder and adjust the displayed absorbance to a reading of 0.000. The extracted dyebath sample, placed in the second holder, was then moved into position and the displayed absorbance was recorded. After repeating this procedure at other applicable wavelengths, the cuvette containing the extracted dyebath sample was removed from the spectrophotometer and the sample either discarded or poured back into the sample vial for storage. The cuvette was then cleaned out and dried. It is very important in making absorbance measurements that the cuvettes be kept scrupulously clean and that entrained air bubbles not be permitted to distort absorbance readings.

## DEVELOPMENT OF DYESTUFF ABSORBANCE CHARACTERISTICS

### Absorbance Curves

As noted previously, dyebaths with more than one dyestuff require absorbance measurements at several wavelengths to enable calculation of component dye concentrations. The wavelengths selected for these measurements are generally those at which one of the dyes has a maximum in absorbance and the other dyes have relatively low absorbances. To determine the absorbance curve for each dye, a standard solution of known concentration is first prepared by adding a measured quantity of dye to a mock dyebath solution. The absorbance of this dye standard is measured at every 10 nanometers wavelength from 380 nm to 700 nm and the data are plotted. This procedure is repeated for every dye in the mixture.

Two styles of carpet were dyed in the study at Mill C-2. Shades for Style 1 were made up of varying amounts of the same three dyestuffs - an orange, a red, and a blue. Style 2 used these same three dyestuffs for some shades, and the same orange with different red and blue dyestuffs for other shades. Table IV-1 summarizes these dyestuff mixtures. It should be noted that although the same three dyes were used in the Style 1 shades and the Style 2 - Group A shades, separate mock dyebaths were required for each of the two styles, due to the fact that different quantities of auxiliary chemicals were used for each style.

Absorbance measurements for the constituent dyes of each of the three dyestuff combinations in Table IV-1 are plotted as absorbance curves in Figures IV-1, IV-2, and IV-3. Based on these absorbance curves, the wavelengths of 400 nm, 540 nm, and 640 nm were selected as the wavelengths to be used for absorbance measurements in subsequent

## TABLE IV-1

## DYE COMBINATIONS EVALUATED

|  | Carpet Style 2 |  |
|---|---|---|
| Carpet Style 1 | Group A Shades | Group B Shades |
| Orange #1 | Orange #1 | Orange #1 |
| Red #1 | Red #1 | Red #2 |
| Blue #1 | Blue #1 | Blue #2 |

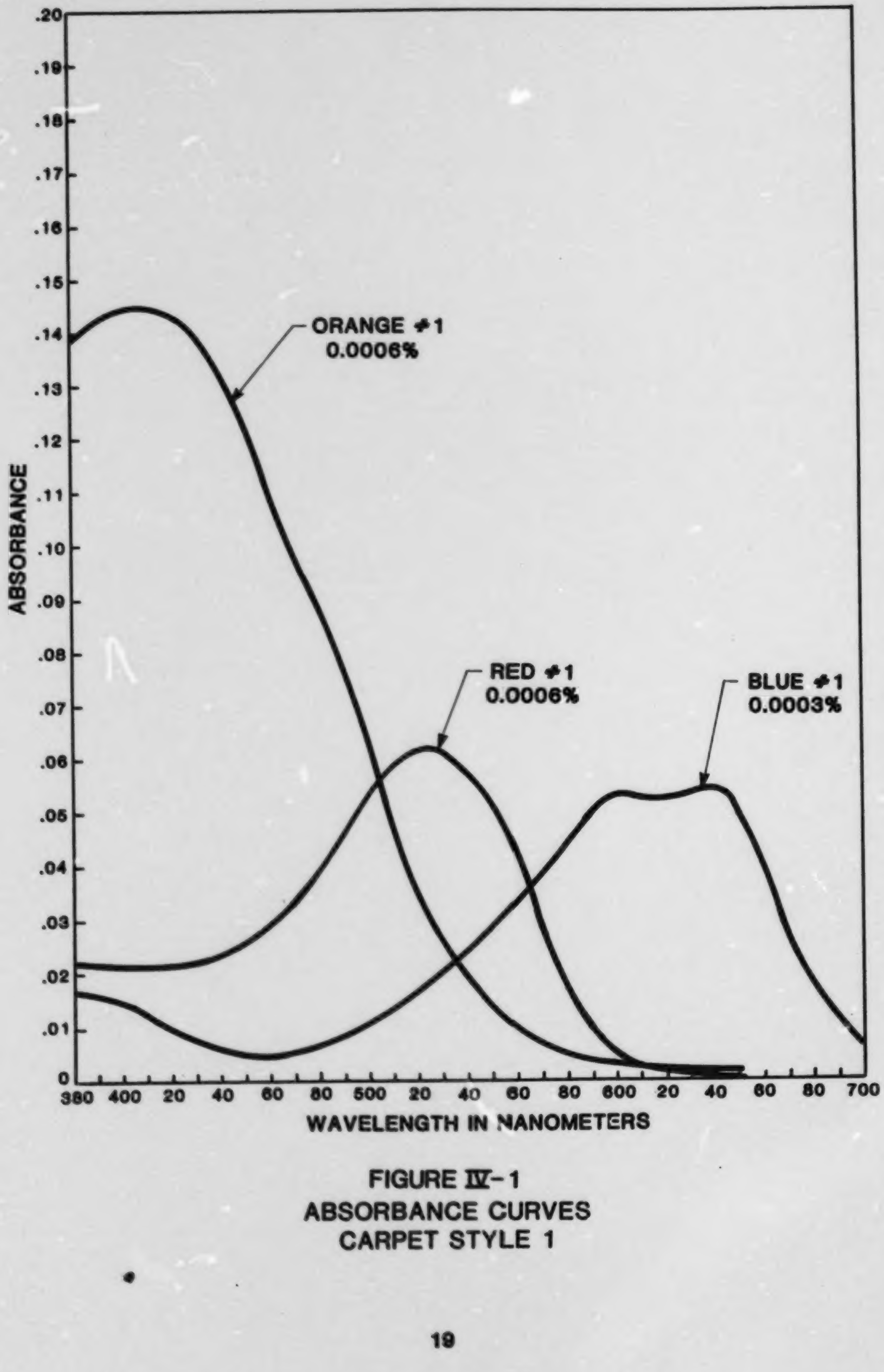

FIGURE IV-1
ABSORBANCE CURVES
CARPET STYLE 1

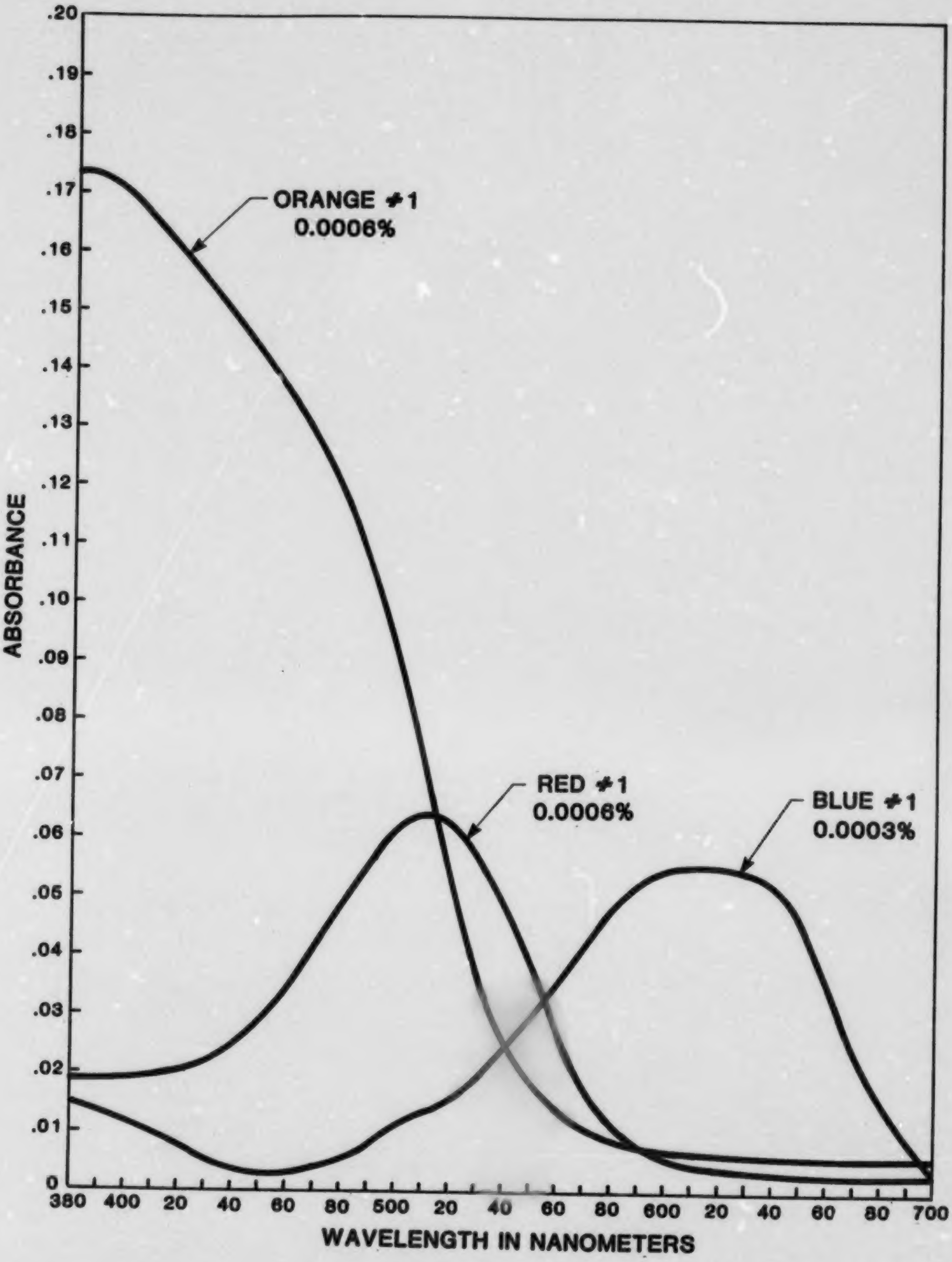

FIGURE IV-2
ABSORBANCE CURVES
CARPET STYLE 2-GROUP A

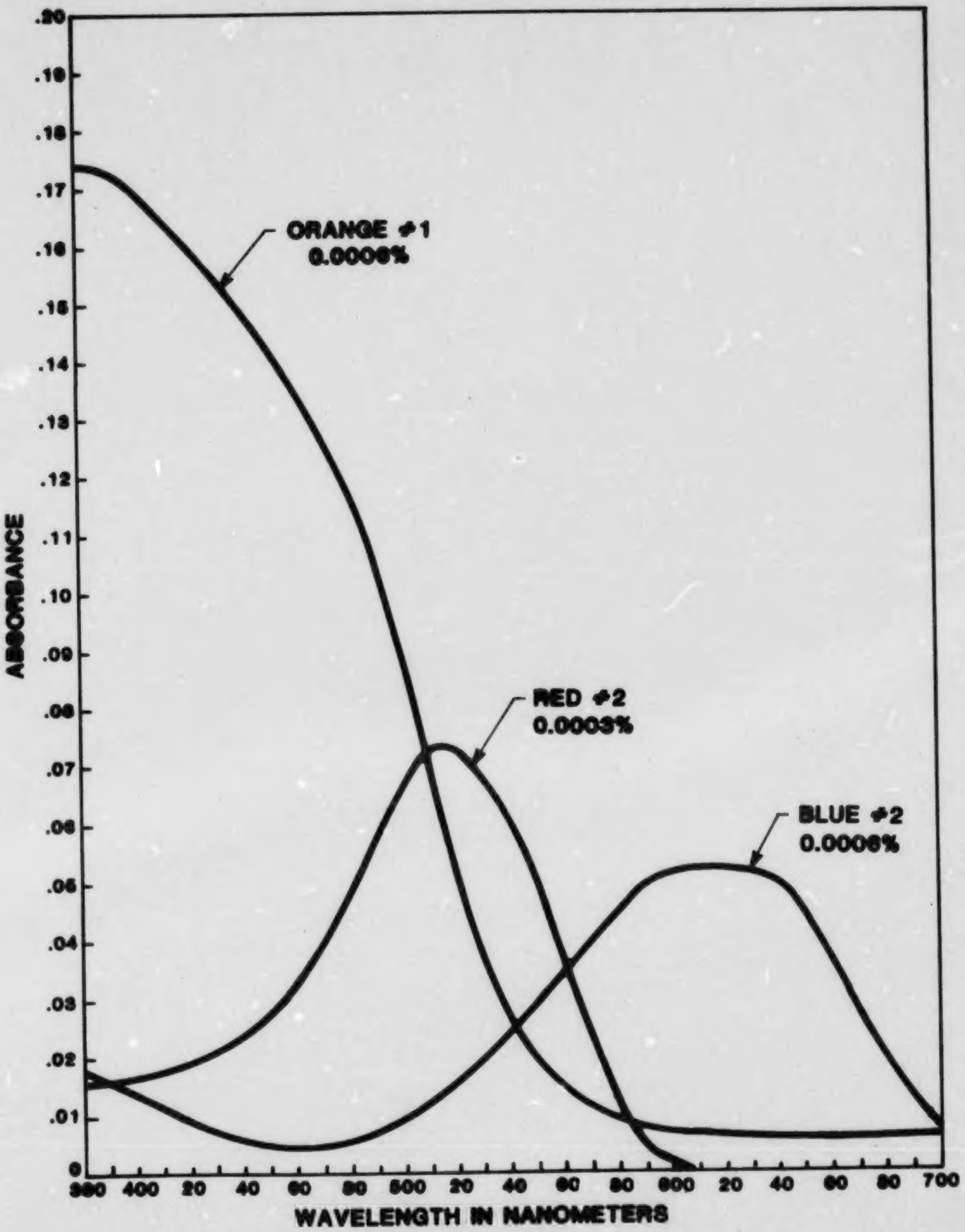

FIGURE III-3
ABSORBANCE CURVES
CARPET STYLE 2-GROUP B

dyebath analyses. At 400 nm, the orange dye has its highest absorbance relative to the red and blue dyes. Thus 400 nm is a good wavelength for analysis. Similarly, at 600 to 640 nm, the blue dye exhibits its maximum absorbance. By selecting a wavelength at the far end, 640 nm, the absorbance from the red and orange dyes is minimized. The selection of the third wavelength is less straightforward. The red dyestuff has its peak absorbance at 520 nm. However, the orange dye is also absorbing light very heavily at this wavelength. By shifting to 540 nm, the absorbance of the red dyestuff decreases only slightly whereas the absorbance of the orange dye is cut by more than half. Also, the absorbance due to the blue dye is still relatively small at 540 nm.

### Dye Absorbance Constants

Once the optimum wavelengths for absorbance measurements have been selected, dye absorbance constants can be determined. As can be seen by rearranging the Lambert-Beer equation for a single dye at a given wavelength, i.e., $k = A/C$, the dye absorbance constant is the slope of absorbance measurements versus known concentrations. Solutions of several known concentrations are prepared for each dye in its mock dyebath. Absorbance measurements of these solutions are made at the previously determined wavelengths. The slope of the absorbance versus concentration data for each wavelength is then determined. These slopes then become the k-values used in dyebath analysis.

For the dye combinations presented in Table IV-1, this procedure was followed using five different concentrations of each dye. Samples were prepared by extraction with octanol, and the absorbance read at 400 nm, 540 nm, and 640 nm. The k-values were determined as the slope of the best linear fit to the absorbance vs. concentration data. The following three sets of simultaneous linear equations result at the wavelengths selected:

Style 1

(at 400 nm) $A = 216.27\ C_o + 24.30\ C_r + 47.20\ C_b$
(at 540 nm) $A = 21.55\ C_o + 102.33\ C_r + 78.18\ C_b$
(at 640 nm) $A = 0.0\ C_o + 0.12\ C_r + 207.15\ C_b$

#### Style 2 - Group A

(at 400 nm) $A = 200.58\ C_o + 23.67\ C_r + 45.71\ C_b$
(at 540 nm) $A = 19.06\ C_o + 98.96\ C_r + 78.22\ C_b$
(at 640 nm) $A = 0.0\ C_o + 0.24\ C_r + 200.13\ C_b$

#### Style 2 - Group B

(at 400 nm) $A = 200.58\ C_o + 59.97\ C_r + 18.70\ C_b$
(at 540 nm) $A = 19.06\ C_o + 248.71\ C_r + 36.83\ C_b$
(at 640 nm) $A = 0.0\ C_o + 0.23\ C_r + 85.25\ C_b$

where: $A$ = absorbance of the dyebath sample at the given wavelength
$C_o$ = concentration of the orange dye (weight percent)
$C_r$ = concentration of the red dye (weight percent)
$C_b$ = concentration of the blue dye (weight percent)

The three coefficients presented in each of these equations are the k-values for the represented dye at the given wavelength.

The accuracy of the k-values in any set of simultaneous equations can be assessed by mixing known concentrations of each dye in mock dyebaths, measuring the absorbance values at each of the relevant wavelengths, calculating dyestuff concentrations by simultaneous solution of the equations, and comparing these calculated concentrations to the known concentrations. Several such mixtures were made and tested. The calculated concentrations were generally within 10 percent of the known concentration. This degree of accuracy is considered acceptable.

## ANALYSIS AND RECONSTITUTION OF EXHAUSTED DYEBATHS

### Determination of Residual Dye Concentrations

Analysis of exhausted dyebaths can be performed after k-values have been established. As noted above, these analyses are performed by measuring the absorbance of the dyebath at selected wavelengths and solving simultaneous linear equations to determine concentrations. Programmable calculators that are capable of solving matrices simplify the solution of these equations. A Hewlett-Packard HP-15C was used in the early stages of this project to do this. Desk-top computers can also be programmed to use absorbance information to calculate residual concentrations and make-up amounts; Section V presents a description of this approach.

## Calculation of Dye and Auxiliary Chemical Make-Up Quantities

Dye Make-up Quantities. Dyestuff quantities required to reconstitute dyebaths in this project were determined in the following manner. The calculated concentration of a given dyestuff was multiplied by the mass of dyebath to be reused to give the total quantity of the dye being reused. This quantity was then subtracted from the total quantity of that dye required by the recipe for the next shade to be dyed; this difference was the amount of dyestuff added as make-up for the next dyeing.

Auxiliary Chemical Make-up Quantities. A detailed and very accurate analysis of the auxiliary chemical quantities remaining in the dyebath is not needed nor would it be easy to obtain. Instead, the amounts of auxiliary chemicals required to be added to reconstitute the dyebath can be estimated to sufficient accuracy by using a simple mass balance equation. The quantity of auxiliary chemicals originally added to a dyebath remains in the exhausted dyebath after dyeing. If all of the exhausted dyebath is reused, then no make-up auxiliary chemicals are required. If only a portion of the exhausted dyebath is reused, then the percent auxiliary chemical make-up is calculated as follows:

$$P = 100 \left[ 1 - \frac{V_r}{V_f} \right]$$

where $P$ = percent make-up of auxiliary chemicals
$V_r$ = volume of dyebath reused
$V_f$ = final volume of dyebath (at time of dye analysis)

In the case where the dyebath has been diluted by overflow rinsing, $V_f$ is the sum of the dyebath volume at the end of the hold cycle (immediately after dyeing) plus the volume of cooling water added.

In most mills, these volumes remain relatively constant and thus the auxiliary chemical make-up percentage need be calculated only once, and revised when dyeing procedure changes are made.

Certain auxiliary chemicals will completely exhaust or evaporate during dyeing and the percent make-up will thus be 100 percent. Examples are defoamers, volatile chemicals, and finishes that adsorb onto the textile fibers.

## SECTION V

## DATA PROCESSING

The reconstitution and reuse of dyebaths requires the following data analysis and processing steps to be performed:

1. Solution of three or more simultaneous linear equations, based on absorbance data, to determine dyestuff concentrations.
2. Conversion of these concentrations to masses of dyestuff in the recycled dyebath.
3. Computation of make-up quantities for the next dyeing based on recipe quantities, recycled quantities, and estimated auxiliary chemical exhaustion rates.

These steps must be performed quickly and accurately to maintain a normal production schedule incorporating dyebath reuse. Because of the complexity of the calculations and the need for accuracy and speed in performing them, an automatic data processing system, based on a desk-top computer, was developed. This system was tested during the pilot-scale and full-scale dyeings.

Computerized systems can be designed to offer a great deal of automation. For example, a highly automated system could be envisioned wherein the computer would control the wavelength setting of the spectrophotometer, automatically collect absorbance data from the spectrophotometer, calculate the make-up quantities, and control metering pumps to add these quantities to the dyebeck for the next dyeing. However, most mills in the textile industry use manual weighing of dyes and chemicals; thus a simple printout of make-up quantities was deemed sufficient. Furthermore, the control of wavelength selection by the computer would add significantly to cost of the spectrophotometer. Therefore, the system developed in this study requires the operator to manually adjust the wavelength and zero the instrument.

The basic components of the data processing system are described below, and their interrelationship is shown in Figure V-1. The operator, through the keyboard, instructs the central processing unit (CPU) to begin dyebath reuse data processing. The CPU, through the

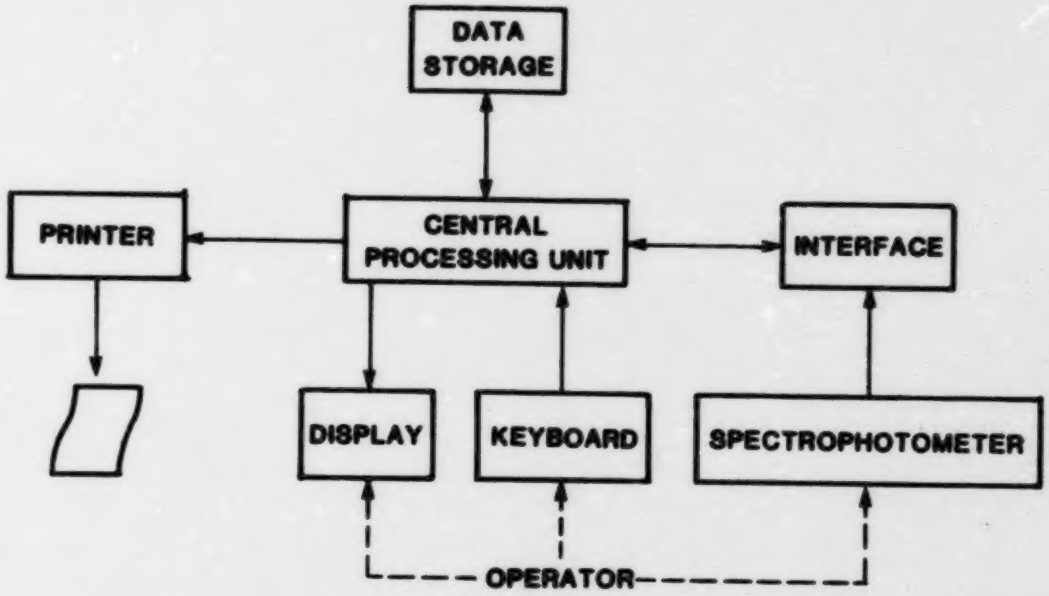
DATA STORAGE
PRINTER
CENTRAL PROCESSING UNIT
INTERFACE
DISPLAY
KEYBOARD
SPECTROPHOTOMETER
OPERATOR

FIGURE V-1
AUTOMATIC DATA PROCESSING SYSTEM

display (CRT screen), requests information from the operator, such as the product style, shade, weight, etc. The operator responds to these questions by typing his responses on the keyboard. At the appropriate times, the CPU instructs the interface to collect absorbance data from the spectrophotometer and transmit these data to the CPU. The CPU processes these data, searches its storage files for the appropriate dyeing recipes, calculates the required make-up quantities and prints the results on the printer. Further details on the individual components and the computer programs used in this study are presented below.

## HARDWARE

The following equipment was used in this study for data analysis:

- o Zenith Data Systems Z-100 computer, including CPU, display, keyboard, and data storage systems.
- o Epson MX-80 printer.
- o Analytical Computers, Inc. Smartface analog-to-digital interface.

The Bausch & Lomb Spectronic 21-DV spectrophotometer used in this study was previously described in Section IV. Similar models produced by other manufacturers could be substituted for the items listed above. More details on the necessary equipment specifications are contained in Volume II of this report.

### Central Processing Unit (CPU)

The Zenith Z-100 actually has two CPU's; an 8088 for 16-bit software and an 8085 for 8-bit software. For this project, 16-bit software was used for the following reasons. Sixteen-bit systems are becoming prevalent, especially among small business computers. Sixteen-bit CPUs generally operate faster and can address more storage memory than 8-bit systems can. This allows more sophisticated programs to be written.

### Data Storage

Two types of data storage are provided with the Zenith Z-100. Temporary storage of the programs and data generated during the calculation process is accomplished with 128k bytes of internal random access memory (RAM). Approximately 100k are required for the dyebath reuse software. This memory is lost whenever power to the computer is turned off.

Permanent data storage is afforded by 5-1/4 inch floppy disk drives. The Zenith Z-100 has two such drives. Each disk for the drives can store 320k bytes of data. If 100k bytes are required for software storage, this leaves 220k bytes for storage of dye recipes. This amount of storage is adequate for several thousand dye recipes. Thus, the entire data set required for dyebath reuse at a typical mill can be stored on a single 5-1/4 inch disk.

### Input-Output Devices

Video Display. The video display is the primary means for transferring information to the operator. Instructions and requests for information are displayed on a video CRT (cathode ray tube). The Zenith Z-100 uses a 12-inch-diagonal screen with capacity for 24 lines of 80 characters.

Keyboard. The keyboard is the primary means for transferring information from the operator to the computer. The operator types in responses to the computer's questions and hits the "return" key to transmit the typed information.

Printer. The printer provides a permanent readible copy of information and thus is useful for printing a copy of the make-up quantities needed for the next dyeing. This copy can then be sent to the drug room for use in weighing the appropriate quantities of chemicals. The printer used for this project is an Epson MX-80 dot matrix printer that is capable of printing 80 characters per second on either standard 8-1/2 x 11-inch paper or on standard 9-1/2 inch wide continuous computer forms. Connection of the printer to the computer is with a standard 25-pin cable to a Centronics compatible parallel printer port on the computer.

Interface. The standard analog voltage output of most laboratory equipment, such as the Spectronic 21-DV (0 to 1 volt), is not in a form that can be interpreted by most computers. Thus, conversion of this analog voltage to a digital (binary) signal is required. This is performed by an analog-to-digital interface. Most computers have an RS-232C input/output port for transmission or receipt of digital data in a serial format. Therefore, it was decided that the best approach to interfacing the spectrophotometer and computer would be to convert the analog output of the spectrophotometer to an RS-232C compatible stream of serial data. For this project, an Analytical Computers, Inc. Smartface interface was selected. The Smartface is a microprocessor controlled interface with a 12-bit (resolution of 1 part in $2^{12}$ or 4096) analog to digital converter, and is used to transfer information in a binary coded decimal (BCD) serial format at a rate of 1,200 bits per second. A standard 25-pin cable is used to connect the interface to the computer, and a simple two-wire cable is used to connect the interface to the recorder output jacks of the spectrophotometer.

## SOFTWARE

Three basic pieces of software are required for the dyebath reuse system: the computer operating system, the computer language interpreter/compiler, and the actual dyebath reuse computer programs.

### Operating System

The most popular 16-bit operating system is MS-DOS from Microsoft, Inc. The version supplied with the Zenith Z-100 is known as Z-DOS. The operating system is basically a program that manages the computer and its peripheral devices (keyboard, display, internal memory, disk drives, input/output ports). It directs and tracks the flow of information between the CPU and various devices, and also provides utility programs for such tasks as copying, editing, and erasing data storage files.

### Language Interpreter/Compiler

Many computer languages have been developed for computers. Popular languages include BASIC, FORTRAN, and COBOL. They are popular because they are machine independent, i.e., they can be transferred from one computer type to another with either no or only very slight modification. Another reason for their popularity is that they are high-level languages, i.e., they direct the computer to do many computations with a single command rather than detailing every step in a form directly understood by the computer (machine language).

BASIC is a readily learned, easily understood computer language. It is also the most widely available programming language for desk-top computers. For these reasons, it was chosen for use in developing the dyebath reuse program.

The computer must convert the BASIC language instructions into a machine language code that it can understand. This is performed with either an interpreter or a compiler. The distinction between interpreter and compiler is not significant for this application; either is acceptable. The version of the BASIC language used for this project is MS-BASIC by Microsoft, Inc. This BASIC interpreter is very popular, powerful, and widely available for 16-bit computers. MS-BASIC is provided with the Z-100 computer as Z-BASIC.

### Dyebath Reuse Program

A listing of the dyebath reuse program is provided in Appendix A. Details on the use of the program are provided in Volume II. A brief description of the dyebath reuse program is provided below.

When the computer is turned on, the CPU automatically executes a simple program, called a boot program. The boot program directs the computer to load the operating system (Z-DOS) from the disk drive into the computer's internal memory. Once in memory, the operating system takes control and automatically executes a series of commands which load first the BASIC interpreter from disk into memory and then the dyebath reuse program. Execution of the dyebath reuse program also begins automatically, and the information in Figure V-2 appears on the display. On this figure and those that follow, the text displayed by the computer

```
              DYEBATH RECONSTITUTION SYSTEM

                      VERSION 3.0


         A.   REUSE DYEING

         B.   UPDATE RECIPES-DATA FILES

         C.   EXIT

     ENTER CHOICE <A, B, or C> A
```

FIGURE
IV-2

is in bold type, while the operator's responses are in normal but underlined type.

The operator chooses option "A" and the computer then prompts for information concerning the last dyeing (the dyebath of which is to be reused). See Figure V-3.

The computer then prompts the operator to set the wavelength on the spectrophotometer and take readings. The operator is asked to verify the absorbance reading that the interface has transferred to the computer. See Figure V-4.

The computer then calculates the quantities of dyestuffs remaining in the exhausted dyebath and reports this information along with the percent dyestuff exhaustion to the operator. See Figure V-5. This information is useful in evaluating whether any problems occurred in the last dyeing. Such problems would be indicated by low dyestuff exhaustions and could be due to pH changes or other causes.

The system then prompts the operator for data on the next dyeing, and asks if any auxiliary chemicals were added in the previous dyeing. See Figure V-6. Finally the computer prompts for information on the assumed exhaustion rates for auxiliary chemicals. In this case, the operator responds that all auxiliary chemicals are exhausted 25 percent except for the defoamer. He assumes that all the defoamer is lost in the previous dyeing. See Figure V-7.

The computer now has all the information it needs to compute and print the make-up quantities for the next dyeing. An example printout is shown in Figure V-8. The format of this printout can be varied to suit a specific mill. The format could also be tailored so that make-up quantities could be printed directly on standard preprinted forms used by the mill.

Further examples on program use are contained in Volume 2, including procedures for storing and updating dye recipes.

```
INPUT LAST STYLE AND SHADE DYED

 1    STYLE #1
 2    STYLE #2
 3
 4
 5
 6
 7
 8
 9
 10
TYPE NUMBER OF STYLE 1

INPUT SHADE NUMBER 33312

STYLE #1  SHADE 33312

CORRECT? <Y or N>  Y

INPUT WEIGHT OF LAST DYEING (Pounds) 2200

 2200 Pounds, Correct? <Y or N>  Y


INPUT VOLUME OF DYEBATH TO BE REUSED (gallons) 5000

 5000 Gallons, Correct? <Y or N>  Y


INPUT TOTAL VOLUME OF EXHAUSTED DYEBATH
INCLUDING ANY OVERFLOW LOSSES (Gallons)  6670
```

**FIGURE**
**V-3**

```
SET WAVELENGTH TO 400 nm

INSERT 'BLANK' CUVETTE AND SET ABSORBANCE AT 0.000
HIT 'RETURN' WHEN READY TO CONTINUE


INSERT SAMPLE CUVETTE
HIT 'RETURN' WHEN READY


ABSORBANCE AT 400  nm = 0.006
CORRECT? <Y or N>  Y
```

```
SET WAVELENGTH TO 540 nm

INSERT 'BLANK' CUVETTE AND SET ABSORBANCE AT 0.000
HIT 'RETURN' WHEN READY TO CONTINUE


INSERT SAMPLE CUVETTE
HIT 'RETURN' WHEN READY


ABSORBANCE AT 540  nm = 0.011
CORRECT? <Y or N>  Y
```

```
SET WAVELENGTH TO 640 nm

INSERT 'BLANK' CUVETTE AND SET ABSORBANCE AT 0.000
HIT 'RETURN' WHEN READY TO CONTINUE


INSERT SAMPLE CUVETTE
HIT 'RETURN' WHEN READY


ABSORBANCE AT 640  nm = 0.004
CORRECT? <Y or N>  Y
```

FIGURE
V-4

```
DYE            CONC %          EXHAUST %
~~~            ~~~~~~          ~~~~~~~~~
ORANGE           1.343464E-05     99.4
RED              8.994066E-05     96.4
BLUE             1.925705E-05     99.2
```

FIGURE
V-5

```
INPUT NEXT STYLE AND SHADE

 1    STYLE #1
 2    STYLE #2
 3
 4
 5
 6
 7
 8
 9
 10
TYPE NUMBER OF STYLE  1

INPUT SHADE NUMBER  33314

STYLE #1  SHADE 33314

CORRECT? <Y or N>  Y

INPUT WEIGHT OF NEXT DYEING (Pounds)   2200

 2200 Pounds, Correct? <Y or N>  Y




WERE ANY AUXILIARY CHEMICALS ADDED DURING
THE PREVIOUS DYE CYCLE ?  N
```

FIGURE
V-6

```
THE SYSTEM WILL ASSUME A 25 % LOSS FOR ALL AUXILIARIES.
DO YOU WISH TO CHANGE THIS TO A DIFFERENT LOSS PERCENTAGE
<Y or N> ?  N
```

```
THE SYSTEM WILL ASSUME THE FOLLOWING LOSSES FOR THE
AUXILIARY CHEMICALS IN THE LAST DYEBATH:

 1 M.S.P.    25 %
 2 T.S.P.    25 %
 3 Sequest.  25 %
 4 Level   A 25 %
 5 Level   B 25 %
 6 Defoamer  25 %
 7           25 %

TO MAKE ANY CHANGES, TYPE NUMBER OF AUXILIARY CHEMICAL
HIT RETURN TO CONTINUE  6
TYPE NEW LOSS PERCENTAGE <0 to 100> 100
```

```
THE SYSTEM WILL ASSUME THE FOLLOWING LOSSES FOR THE
AUXILIARY CHEMICALS IN THE LAST DYEBATH:

 1 M.S.P.    25 %
 2 T.S.P.    25 %
 3 Sequest.  25 %
 4 Level   A 25 %
 5 Level   B 25 %
 6 Defoamer  100 %
 7           25 %

TO MAKE ANY CHANGES, TYPE NUMBER OF AUXILIARY CHEMICAL
HIT RETURN TO CONTINUE
```

FIGURE
V-7

```
                              EPA - IERL
                           CARPET MILL C-2
                          SVERDRUP & PARCEL

                                                                   10-14-1983
                                                                     10:08:55

LOT:_______________

PRESENT STYLE: STYLE #1        SHADE: 33314    WEIGHT:   2200 lbs

PREVIOUS:      STYLE #1               33312              2200 lbs

VOLUME RECYCLED: 5000 GALLONS


ADD QUANTITIES (Pounds)

DYESTUFF      RECYCLE    RECIPE     ADD

ORANGE           0.01      1.00    0 lbs, 6952 gr
RED              0.04      1.03    0 lbs, 6975 gr
BLUE             0.01      0.96    0 lbs, 6688 gr

AUXILIARY     RECYCLE    RECIPE     ADD

M.S.P.          16.50     22.00      5.50
T.S.P.          16.50     22.00      5.50
Sequest.         8.25     11.00      2.75
Level  A        16.50     22.00      5.50
Level  B        33.00     44.00     11.00
Defoamer         0.00     22.00     22.00

                           EXPERIMENTAL DATA

WAVELENGTH    ABSORBANCE               DYESTUFF     CONCENTRATION %     EXHAUST %

   400           0.006                  ORANGE       1.343464E-05          99.44
   540           0.011                  RED          8.994066E-05          96.37
   640           0.004                  BLUE         1.925705E-05          99.17
```

FIGURE
V-8

# SECTION VI

# BENCH-SCALE DYEBATH REUSE EXPERIMENTS

This section describes bench-scale dyebath reuse experiments performed at the laboratories of Mill C-2. It is organized as follows. The objectives of these bench-scale experiments are discussed first. Preparatory activities prior to initiating the bench-scale experiments are reviewed. The procedures used in the bench-scale beaker dyeings are next described, followed by a presentation and discussion of the results of these dyeings. This section concludes with an evaluation of the advantages and limitations of bench-scale experiments.

## OBJECTIVES

A principal objective of performing beaker dyeing experiments was to enable project personnel to gain experience in performing dyebath analyses and calculations associated with dyebath reconstitution and reuse. This is easily accomplished through beaker dyeings since several series of dyeings can be conducted simultaneously with minimum expenditure of manpower and materials.

A second objective was to determine quickly whether shade matching could be obtained. Also, if problems existed with initial reconstitution procedures or analytical measures, they could be identified and resolved relatively early in the project.

## PREPARATORY ACTIVITIES

Preliminary meetings were held to discuss the project scope and other details prior to initiation of laboratory activities. The necessity of developing information on dye characteristics, including wavelength selection and k-value development (Section IV), prior to any reuse dyeings was discussed. Participants in the study agreed that several weeks of laboratory-scale (beaker) and pilot-scale (beck) trials should be performed prior to full-scale dyeings at the mill. Two contrasting styles of nylon carpet were selected for use throughout these reconstitution trials; Style 1 is a low pile, low grade carpet while Style 2 is a high pile, high grade carpet. In addition, it was decided that beaker dyeings and pilot-scale beck dyeings would be performed using six shades from each of the two styles. The formulation of these twelve shades is presented in Table VI-1.

Fifteen reconstitution series were planned at the beaker dyeing level to achieve the objectives of these bench-scale experiments. Twelve of these were single shade series. Each of the twelve shades was to be dyed five times consecutively, using reconstituted baths, to determine the level of reproducibility and shade matching obtained. The final three series were to be multi-shade series, to see how closely the individual dyeings from a multi-shade series corresponded to the shade standards for each shade.

## PROCEDURES

Laboratory beaker dyeings are commonly employed by Mill C-2 to develop new shades, determine dye formulations, or provide other experimental information. In general, the purpose of beaker dyeing is to simulate mill dyeings, albeit on a much smaller scale. With the exception of modifications required for this study to employ dyebath reconstitution and reuse, the mill's typical beaker dyeing procedures were followed; steps 1 through 6 in the procedures outlined below are similar to those used for most other beaker dyeing activities at Mill C-2. (A few aspects of the dyeing procedures below are asterisked; these items represent procedural refinements made subsequent to preliminary difficulties.) These reconstitution trial procedures are as follows:

Step 1. Add appropriate volume of stock dye solution and 40 ml auxiliary chemical solution to 1000 ml stainless steel beaker and fill with tap water to 800 ml (first dyeing only).

Step 2. Measure pH, and if not 6.5 ± 0.2 for Style 1, or 8.0 ± 0.2 for Style 2, adjust pH to appropriate range by addition of monosodium phosphate (MSP) or trisodium phosphate (TSP).

## TABLE VI-1

## BEAKER- AND PILOT-SCALE DYEING SHADES AND FORMULATIONS

Carpet Style 1

| | Dyestuffs (% owf*) | | | | | |
|---|---|---|---|---|---|---|
| Dyestuff | Shade 101 | Shade 102 | Shade 103 | Shade 104 | Shade 105 | Shade 106 |
| Orange #1 | .04 | .05 | .05 | .30 | .54 | .64 |
| Red #1 | .02 | .02 | .05 | .25 | .55 | .37 |
| Blue #1 | .01 | .01 | .04 | .04 | .05 | .12 |

| Dyebath Auxiliary Chemicals (% owf) | |
|---|---|
| MSP | 1.5 |
| Sequestering Agent | 0.25 |
| Levelers | 2.5 |

Carpet Style 2

| | Dyestuffs (% owf) | | | | | |
|---|---|---|---|---|---|---|
| Dyestuff | Shade 201 | Shade 202 | Shade 203 | Shade 204 | Shade 205 | Shade 206 |
| Orange #1 | .028 | .08 | .11 | .11 | .35 | .97 |
| Red #1 | .019 | .07 | .10 | .16 | -- | -- |
| Blue #1 | .007 | .03 | .07 | .05 | -- | -- |
| Red #2 | -- | -- | -- | -- | .12 | .17 |
| Blue #2 | -- | -- | -- | -- | .25 | .18 |

| Dyebath Auxiliary Chemicals (% owf) | |
|---|---|
| MSP | 0.5 |
| TSP | 1.0 |
| Sequestering Agent | 0.25 |
| Levelers | 2.0 |

* Percent by weight of fiber weight.

Step 3. Place 40 grams of carpet into reciprocating basket and drop basket into beaker.

Step 4. Raise dyebath temperature to boiling.

Step 5. Boil light shades for 30 minutes, medium shades for 45 minutes, and dark shades for 60 minutes. Closely monitor volume and add hot tap water to replace any significant evaporation loss.*

Step 6. Shut heat off, raise basket, and allow carpet to drip for five minutes. Dry carpet sample and measure color characteristics of carpet with Diano/Hardy II color spectrophotometer.

Step 7. Pour exhausted dyebath into graduated cylinder and record volume. Increase (or decrease) volume to 700*ml. Remove 50*ml sample for absorbance measurements. Return remaining (650*ml) exhausted dyebath to beaker and measure pH.

Step 8. Perform filtration or solvent extraction of exhausted dyebath sample.

Step 9. Make absorbance measurements, determine amount of dye remaining in dyebath, and calculate amount required for subsequent dyeing.

Step 10. Add calculated quantity of dye, 5 ml of auxiliary chemical solution, and tap water to bring dyebath volume in beaker to 800 ml.

(Repeat Steps 2 through 10 for subsequent dyeings in series.)

## RESULTS

Fifteen beaker dyeing reconstitution series were performed. Twelve of these series were repetitive dyeings of a single shade. Two Style 1 and one Style 2 multi-shade series were also performed. Most of these series consisted of five dyeings before the dyebath was discarded. A summary of these dyeings is presented in Table VI-2.

### Preliminary Difficulties

As seen in Table VI-2, dyebath samples from the initial beaker dyeings were prepared for analysis using filtration. Samples from several beaker dyeing series were prepared using both filtration and

## TABLE VI-2

## BEAKER DYEING SUMMARY

| Series Number | Carpet Style | Number of Dyeings | Shade Dyeing Sequence | Method of Dyebath Analysis* |
|---|---|---|---|---|
| 1 | 1 | 5 | 105-105-105-105-105 | F |
| 2 | 1 | 5 | 106-106-106-106-106 | F |
| 3 | 1 | 5 | 103-103-103-103-103 | F |
| 3A | 1 | 5 | 103-103-103-103-103 | E, F |
| 4 | 1 | 5 | 104-104-104-104-104 | F |
| 5 | 1 | 4 | 102-102-102-102 | E, F |
| 6 | 1 | 5 | 101-101-101-101-101 | E, F |
| 7 | 2 | 5 | 205-205-205-205-205 | E, F |
| 8 | 2 | 5 | 206-206-206-206-206 | E, F |
| 9 | 2 | 5 | 204-204-204-204-204 | E, F |
| 10 | 2 | 5 | 202-202-202-202-202 | E, F |
| 10A | 2 | 2 | 202-202 | E |
| 11 | 2 | 5 | 203-203-203-203-203 | E, F |
| 12 | 2 | 5 | 201-201-201-201-201 | E, F |
| 13 | 2 | 6 | 201-202-203-204-203-202 | E |
| 14 | 1 | 5 | 101-102-103-104-105 | E |
| 15 | 1 | 5 | 106-104-103-101-101 | E |

* F = Sample preparation by filtration.

E = Sample preparation by solvent extraction.

extraction. Subsequently only extraction was employed for sample preparation. This pattern was the result of difficulties experienced in the initial dyeings. The initial beaker dyeings were attempted using Style 1 carpet swatches. The reconstituted dyeings of the dark shades, and to some extent the medium shades, were successful. This encompassed series 1 to 4. However, problems with reconstituting the dyebaths of light shades (series 5 and 6) were immediately obvious. Analysis of the exhausted dyebaths implied that very poor dyestuff exhaustion had occurred. In actuality, the dyestuffs had exhausted very well. Thus, smaller than necessary amounts of make-up dye were added for the next dyeing, resulting in a light dyeing.

The difficulties experienced in these initial dyeings were the result of a number of factors. In retrospect, the principal factors appear to have been dirty cuvettes and inconsistent dyeing procedures. At the time, however, some aspect of the filtration procedure used for sample preparation itself was suspected. Consequently, a switch was made to solvent extraction for preparing exhausted dyebath samples for analysis. In addition, the cuvettes were cleaned after each analysis with solvent to remove any buildup. And, a rigorous, consistent procedure as presented above was instituted to eliminate any variations due to dyebath volume changes caused by evaporation. These modifications resulted in successful reconstitution series for the two light shades.

Subsequently, reconstitution series using Style 2 carpet swatches were performed (series 7 to 12). For comparative purposes, exhausted dyebath samples from these dyeings were prepared for analysis by both approaches, filtration and solvent extraction. This comparison, shown as percent dyestuff exhaustion by procedure for each of the three dyes in the exhausted baths, is presented in Table VI-3. These results illustrate that slightly lower absorbances, i.e., lower concentrations or higher exhaustions of dyes, were measured in samples prepared using solvent extraction. Apparently either minute amounts of interfering substances remain in the filtered samples, or small amounts of dyestuff are lost from the extracted samples. Regardless, under controlled

## TABLE VI-3

## COMPARISON OF ANALYTICAL PROCEDURES

| | | Percent Exhaustion | | | | | |
|---|---|---|---|---|---|---|---|
| | | Orange Dye | | Red Dye | | Blue Dye | |
| Series Number | Bath Number | Extraction | Filter | Extraction | Filter | Extraction | Filter |
| 7 | 1 | 95.2 | 94.9 | 97.0 | 95.9 | 92.6 | 96.0 |
| 7 | 2 | 94.6 | 93.4 | 96.5 | 94.3 | 92.5 | 95.5 |
| 7 | 3 | 94.9 | 90.9 | 96.5 | 93.3 | 94.2 | 94.7 |
| 7 | 4 | 95.6 | 92.1 | 97.5 | 95.2 | 95.0 | 96.2 |
| 7 | 5 | 95.7 | 91.5 | 97.4 | 94.8 | 94.8 | 95.7 |
| 8 | 1 | 91.3 | 91.5 | 95.0 | 93.2 | 87.2 | 93.2 |
| 8 | 2 | 90.1 | 89.3 | 93.8 | 88.8 | 87.6 | 91.9 |
| 8 | 3 | 90.7 | 88.7 | 94.7 | 89.3 | 88.8 | 92.0 |
| 8 | 4 | 89.0 | 86.5 | 93.5 | 86.6 | 88.5 | 90.7 |
| 8 | 5 | 87.6 | 85.1 | 92.4 | 85.9 | 85.2 | 89.3 |
| 9 | 1 | 93.5 | 94.6 | 94.0 | 93.7 | 94.9 | 94.0 |
| 9 | 2 | 97.3 | 93.7 | 96.0 | 95.3 | 96.7 | 95.5 |
| 9 | 3 | 96.0 | 91.9 | 96.7 | 94.8 | 93.7 | 95.1 |
| 9 | 4 | 97.6 | 90.5 | 96.9 | 95.0 | 97.4 | 94.9 |
| 9 | 5 | 97.5 | 90.0 | 97.1 | 95.6 | 97.4 | 95.5 |
| 10 | 1 | 95.9 | 95.9 | 95.0 | 94.8 | 97.3 | 96.9 |
| 10 | 2 | 96.6 | 94.7 | 96.1 | 95.6 | 97.0 | 96.0 |
| 10 | 3 | 97.8 | 93.4 | 97.2 | 95.7 | 97.5 | 96.3 |
| 10 | 4 | 96.3 | 92.8 | 97.9 | 96.2 | 98.4 | 96.3 |
| 10 | 5 | 98.0 | 92.9 | 97.4 | 96.7 | 98.1 | 96.9 |
| 11 | 1 | 94.4 | 94.7 | 91.7 | 92.4 | 93.5 | 93.3 |
| 11 | 2 | 95.9 | 93.1 | 93.2 | 92.0 | 94.4 | 93.9 |
| 11 | 3 | 97.6 | 91.8 | 94.5 | 92.7 | 97.8 | 94.2 |
| 11 | 4 | 96.1 | 91.4 | 95.2 | 93.4 | 96.0 | 94.6 |
| 11 | 5 | 97.4 | 91.0 | 96.4 | 94.4 | 97.3 | 95.6 |
| 12 | 1 | 92.3 | 91.6 | 88.6 | 87.5 | 91.5 | 87.4 |
| 12 | 2 | 95.2 | 91.9 | 93.4 | 91.3 | 95.2 | 93.7 |
| 12 | 3 | 95.4 | 93.6 | 96.4 | 96.5 | 97.6 | 95.0 |
| 12 | 4 | 98.2 | 94.5 | 97.5 | 97.2 | 97.6 | 97.5 |
| 12 | 5 | 97.3 | 92.8 | 97.1 | 97.5 | 96.3 | 96.2 |

conditions, the resulting differences between these two procedures are insignificant. Make-up quantities for this set of dyeings were calculated on the basis of solvent extraction procedure measurements.

### Shade Matching

Although Mill C-2 has a standard reference for each shade and style in their product line, these standards are based on full-scale beck dyeings. Consequently, to evaluate the success of reconstituted beaker dyeings, a separate carpet standard had to be devised. For these experiments, the first beaker dyeing of each of the twelve shades was called the standard, since these first bath carpet samples were dyed conventionally, i.e. in a fresh bath.

Carpet sample color measurements were determined using a Diano/Hardy II spectrophotometer. Each carpet sample was placed in a compression holder. Percent reflectance was measured every twenty nanometers from 400 to 700, for a total of 16 values. This data was used to determine color differences relative to the carpet standard, measured in the same way as the sample. Color differences under conditions simulating daylight, tungsten, and cool, white fluorescent light were developed using the C.I.E. L*a*b* color difference equation (Starlab units) for each illuminant conditon. A Starlab unit can be converted to a MacAdam unit if multiplied by a factor of approximately 2. Only small variations in color differences were observed among the three illuminant conditions.

Table VI-4 presents color difference data for each of the reconstituted beaker dyeings. Color differences for the three illuminant conditions have been averaged for presentation in this table. These color differences give a quantitative measure of the uniformity of the carpet sample dyed in reconstituted baths. It should be noted that, in contrast to full-scale dyeings, no dye adds were attempted during beaker- or pilot-scale trials. In full-scale dyeing, significant color differences are corrected by adding more dye to the dyebath and continuing the dyeing until the shade becomes acceptable.

## TABLE VI-4

## BENCH-SCALE SHADE MATCHING RESULTS

| Series Number | Shade | Average Color Differences (Starlab Units)<br>Bath #1 | Bath #2 | Bath #3 | Bath #4 | Bath #5 | Bath #6 |
|---|---|---|---|---|---|---|---|
| 1 | 105 | Std | 0.78 | 2.16 | 1.55 | 2.52 | -- |
| 2 | 106 | Std | 0.63 | 0.41 | 0.89 | 0.54 | -- |
| 3 | 103 | Std | 5.86 | 3.11 | 3.42 | 3.20 | -- |
| 3A | 103 | Std | 2.29 | 3.13 | 3.74 | 3.32 | -- |
| 4 | 104 | Std | 1.81 | 5.11 | 1.98 | 1.83 | -- |
| 5 | 102 | Std | 0.86 | 1.32 | 2.37 | -- | -- |
| 6 | 101 | Std | 1.10 | 2.08 | 1.28 | 1.60 | -- |
| 7 | 205 | Std | 0.87 | 1.73 | 1.01 | 1.57 | -- |
| 8 | 206 | Std | 1.21 | 1.35 | 1.42 | 1.53 | -- |
| 9 | 204 | Std | 0.58 | 0.52 | 0.92 | 0.60 | -- |
| 10A | 202 | Std | 0.91 | -- | -- | -- | -- |
| 10 | 202 | -- | -- | 1.15 | 1.30 | 0.99 | -- |
| 11 | 203 | Std | 0.09 | 0.67 | 0.34 | 1.58 | -- |
| 12 | 201 | Std | 2.05 | 2.46 | 3.06 | 2.63 | -- |
| 13 | Multi | 0.42 | 1.17 | 5.65 | 2.89 | 4.47 | 3.86 |
| 14 | Multi | 0.95 | 1.06 | 1.98 | 2.37 | N/A | -- |
| 15 | Multi | 1.05 | 2.60 | 1.75 | 2.10 | 1.77 | -- |

N/A - Data not available

### Discussion

A review of Table VI-4 shows that over 80 percent of the reconstituted dyeings had color differences from the standard of less than 3 Starlab units (approximately 6 MacAdam units). These relatively small color differences would normally be corrected in production dyeing by making a dye add. Dyeings performed after institution of procedural modifications, i.e., series 5 through 15, were more successful than the previous dyeings. These beaker dyeings achieved their objectives in showing that good shade matching with dyebath reconstitution and reuse was possible at this mill, and in providing an opportunity to become familiar with, evaluate, and develop necessary reuse and analysis procedures.

## ADVANTAGES AND LIMITATIONS OF BEAKER DYEINGS

Beaker dyeings have several advantages as well as severe limitations. These are both discussed below.

### Advantages

The most important advantage of beaker dyeing is that it does not risk carpet. Several thousand beaker dyeings could be performed using the same amount of carpet required for one full-scale dyeing. Therefore, it is a quick and inexpensive approach to evaluate the potential applicability of dyebath reconstitution and reuse to a given dyeing situation.

All activities and monitoring can be performed by a single individual. Also, since several reconstitution series can be performed simultaneously using only a single counter or bench, mill personnel can quickly gain experience and confidence in the analyses, calculations, procedures, and concepts of dyebath reconstitution and reuse. In addition, beaker dyeings allow problems in the approach or procedures to be isolated so that solutions can be developed and implemented.

### Disadvantages

The most severe limitation of beaker dyeing is that it cannot replicate beck dyeing in certain respects. Depending on the particular circumstances at a mill, aspects such as the following may be at variance: method of heating (e.g., hot plate vs. live steam); method of dyebath cooling (e.g., atmospheric vs. displacement with cooling water); method of carpet and dyebath circulation; method of dye and auxiliary chemical addition; method of rinsing; etc. In addition, with the exception of shade matching, levelness and other finished carpet characteristics cannot be evaluated due to the small amount of carpet dyed in beaker dyeings. Even if sufficient carpet sample were available to conduct these tests, the results in these areas would be of little relevance due to the significant dissimilarities between bench- and full-scale dyeing.

These limitations naturally lessen the ability of bench-scale tests to determine the ease or difficulty of achieving successful full-scale reconstitution dyeings. To overcome most of these limitations, pilot-scale beck dyeings were used as a transition between beaker dyeings and full-scale trials.

# SECTION VII

# PILOT-SCALE DYEBATH REUSE EXPERIMENTS

This section describes pilot-scale activities performed at the laboratories of Mill C-2. The format is similar to that of Section VI. The objectives of these pilot-scale experiments are presented first. Preparatory activities prior to initiating pilot-scale beck dyeings are reviewed, and procedures used in these dyeings are described. The results obtained in these pilot-scale reconstitution experiments are next presented and discussed. The section concludes with an evaluation of the advantages and limitations of these pilot-scale dyeings.

## OBJECTIVES

For all of its advantages as a test method, the applicability of beaker dyeing results to predict the outcome of full-scale reconstitution trials is limited, as noted in the previous section. Therefore, a principal objective of pilot-scale beck dyeing is to simulate full-scale dyeing as closely as possible. As with beaker dyeings, an important concern was to identify and resolve any problems that developed, particularly if they involved dyed carpet characteristics. In this regard, related objectives were to determine acceptable temperatures for beginning a dyeing in a recycled dyebath, and any constraints on the sequence of shades that can be dyed in a series.

## PREPARATORY ACTIVITIES

Eight reconstitution series were planned to achieve the objectives listed above. Six shades (three for each carpet style) were to be dyed five times consecutively using reconstituted baths to determine the level of reproducibility and shade matching obtained. In addition, a ten-bath multishade series of each carpet style was planned to evaluate typical dyebath reuse sequences.

The piece of carpet dyed in each bath was approximately five yards long by one yard wide. Sufficient greige carpet in the two styles was acquired and cut to the appropriate dimensions; Style 2 carpets were cut shorter than Style 1 carpets to account for their increased face yarn weight. Also, prior to performing any dyeings, the beck was calibrated by adding known volumes of water and measuring the corresponding height on the side of the beck.

## PROCEDURES

As with beaker dyeing, Mill C-2 uses pilot-scale beck dyeing to develop evaluative and experimental information. Again, the purpose of pilot-scale beck dyeing is to simulate full-scale mill dyeings. The typical pilot-scale beck dyeing procedures used by Mill C-2 were followed in this study with the exception of modifications required to employ dyebath reconstitution and reuse. The procedures used in these reconstitution trials were as follows:

Step 1. Weigh carpet piece to be dyed and add water into beck to 20:1 liquor ratio by weight (first bath only).

Step 2. Introduce carpet into beck and start reel.

Step 3. Add auxiliary chemicals across front of beck and run for five minutes.

Step 4. Measure pH, and if not at 7.0 ± 0.2 for Style 1, or 8.0 ± 0.2 for Style 2, adjust pH into appropriate range by addition of MSP or TSP.

Step 5. Add appropriate amount of dyes (mixed in solution) over a five-minute period across the front of the beck.

Step 6. Turn live steam on in beck five minutes after the last of the dye solution has been added. Raise temperature in beck at 3°F per minute to 180°F.

Step 7. Dye carpet for 30 minutes at 180°F; maintain constant volume with necessary additions of hot tap water; maintain pH in required range for style being dyed.

Step 8. Cut steam and lower temperature to 150°F with addition of cold water to front of beck.

Step 9. Stop machine and roll carpet onto reel. Let carpet drip for five to ten minutes.

Step 10. Remove carpet for separate rinsing and drying. Measure exhausted dyebath volume. Collect exhausted dyebath samples for absorbance measurements and environmental analyses.

Step 11. Perform solvent extraction of exhausted dyebath sample.

Step 12. Weigh new carpet piece and drain exhausted dyebath until 20:1 liquor ratio remains for next dyeing.

Step 13. Make absorbance measurements on extracted sample, calculate amounts of dye and auxiliary chemicals remaining in exhausted dyebath, and determine quantities to be added for next dyeing.

Step 14. Raise (or air-cool) exhausted dyebath temperature to 140°F for the start of the next dyeing.

(Repeat Steps 2 through 14 for subsequent dyeings in series.)

## RESULTS

A set of dyeings with varying initial temperatures was conducted first to determine constraints on the initial temperature. Following these dyeings, seven reconstitution series were dyed using the pilot-scale beck. In addition to several five-bath repetitive dyeings of a single shade, a ten-bath multishade series was run for each style. A listing of these dyeings is presented in Table VII-1.

### Temperature Dyeings

Prior to beginning reconstitution dyeings in the pilot-scale beck, several carpet pieces were dyed using conventional procedures. The purpose of these dyeings was to determine the effect of initial dyeing temperature, i.e., temperature at which dye is added, on resultant carpet quality. Initial temperatures of 120°, 140°, 160°, and 180° F were tested. Quantitative shade matching and levelness measurements were not performed. However, visual inspection showed no apparent deterioration in quality at initial dyeing temperatures as high as 180°F for Style 1. Problems in levelness were noted at 180°F for Style 2, but at 140°F and lower, no difficulties were encountered.

## TABLE VII-1

## PILOT-SCALE BECK DYEING SUMMARY

| Series Number | Carpet Style | Number of Dyeings | Shade Dyeing Sequence | Method of Dyebath Analysis** |
|---|---|---|---|---|
| 16 | 1 | 4* | 103/103/103/103 | E |
| 17 | 1 | 5 | 103-103-103-103-103 | E |
| 18 | 1 | 5 | 101-101-101-101-101 | E |
| 19 | 1 | 10 | 101-101-102-103-103-104-104-105-106-106 | E |
| 20 | 2 | 6 | 201-201-201-201-201-201 | E |
| 21 | 2 | 5 | 203-203-203-203-203 | E |
| 22 | 2 | 5 | 206-206-206-206-206 | E |
| 23 | 2 | 10 | 201-201-202-202-203-203-204-203-204-204 | E |

* Four individual dyeings to determine acceptable initial temperature; reconstitution not attempted in Series 16.

** E = Sample preparation by solvent extraction.

The first pilot-scale dyebath reuse experiments used live steam for heating, but it was noted that this caused the dyebath pH to drift significantly from its initial value, resulting in poor dyestuff exhaustion. Use of steam coils for heating eliminated this problem, but resulted in insufficient mixing of the dyebath and resultant side-to-side shade variations. As a result, for all subsequent dyeings live steam was used, and pH was closely monitored and maintained.

### Shade Matching

A description of the equipment and procedures used to perform color measurements on carpet samples is presented in Section VI. The mill's standard reference for a given shade and style was used as the standard for these pilot-scale dyeing measurements. Table VII-2 presents color difference data for the pilot dyeings in reconstitution series 17 through 23. An explanation of the data presented in this table follows. Again, note that no dye adds were performed to correct the dyed carpet shade prior to these measurements.

Four carpet patches were cut from the carpet piece dyed in each bath. These patches were taken 6 inches "in" and 54 inches "down" or "up" from each corner of the piece. Each patch was measured against the standard, and the resultant color differences under the three illuminant conditions have been averaged for each patch. The averages of the four patches from each piece were then themselves averaged to give the values, in Starlab units, shown as the shade matching results.

In addition, the two patches from each end of a carpet piece were compared against each other to determine the amount of side-to-side color variation. The resultant color differences under the three illuminant conditions were averaged for each comparison. Two comparisons were made per carpet piece (i.e., each end), and these two comparisons themselves were averaged for each dyeing to give the values, in Starlab units, shown as side-to-side levelness.

### Color Fastness

The first and last dyeings in series 17 and 22 were tested for color fastness by standard test methods including 60-hour xenon, wet and dry crocking, bleed, and shampoo tests. Neither carpet style was affected in its fastness properties after five dyeings in the same dye liquor.

## TABLE VII-2

## PILOT-SCALE SHADE MATCHING AND LEVELNESS RESULTS

### Shade Matching

| Series Number | Carpet Style | Shade | Bath #1 | Bath #2 | Bath #3 | Bath #4 | Bath #5 | Bath #6 | Bath #7 | Bath #8 | Bath #9 | Bath #10 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 17 | 1 | 103 | 3.08 | 4.11 | 4.64 | 4.28 | 3.41 | -- | -- | -- | -- | -- |
| 18 | 1 | 101 | 1.10 | 2.43 | 3.97 | 1.34 | 1.52 | -- | -- | -- | -- | -- |
| 19 | 1 | Multi | 1.50 | 1.78 | 1.54 | 1.22 | 1.11 | 5.53 | 4.00 | 3.56 | 3.85 | 4.51 |
| 20 | 2 | 201 | 0.83 | 1.17 | 0.93 | N/A | 2.69 | N/A | -- | -- | -- | -- |
| 21 | 2 | 203 | 0.45 | 0.68 | 0.69 | N/A | 1.50 | -- | -- | -- | -- | -- |
| 22 | 2 | 206 | 1.90 | 1.83 | 1.74 | 2.62 | 2.40 | -- | -- | -- | -- | -- |
| 23 | 2 | Multi | 1.66 | 1.04 | 3.78 | 3.80 | 1.66 | 1.21 | 2.75 | 2.50 | 3.10 | 2.95 |

### Levelness

| Series Number | Carpet Style | Shade | Bath #1 | Bath #2 | Bath #3 | Bath #4 | Bath #5 | Bath #6 | Bath #7 | Bath #8 | Bath #9 | Bath #10 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 17 | 1 | 103 | 1.00 | 4.69 | 6.69 | 5.02 | 0.62 | -- | -- | -- | -- | -- |
| 18 | 1 | 101 | 1.58 | N/A | 7.66 | 2.34 | N/A | -- | -- | -- | -- | -- |
| 19 | 1 | Multi | 0.64 | 1.59 | 2.98 | 1.83 | 1.70 | 0.73 | 1.50 | 1.07 | 0.96 | 1.07 |
| 20 | 2 | 201 | 0.80 | 0.94 | 0.70 | N/A | 1.01 | N/A | -- | -- | -- | -- |
| 21 | 2 | 203 | 0.53 | 0.68 | 0.90 | N/A | 0.48 | -- | -- | -- | -- | -- |
| 22 | 2 | 206 | 1.55 | 1.60 | 0.87 | 0.79 | 0.99 | -- | -- | -- | -- | -- |
| 23 | 2 | Multi | 0.86 | 0.90 | 1.81 | 1.88 | 1.76 | 1.72 | 1.49 | 4.08 | 0.76 | 2.00 |

N/A - Data not available.

## Discussion

In general, color differences from standard for all Style 1 dyeings were slightly greater than for Style 2. Also, for both styles, color differences were slightly greater for carpets dyed in reconstituted baths than for those dyed in fresh baths. Nevertheless, all but one of the reconstituted dyeings had a color difference from standard of less than five Starlab units.

Similar trends were observed in the side-to-side measurements. Slightly better results were seen for fresh baths than for reconstituted baths, and for Style 2 dyeings than for Style 1 dyeings. Three-fourths of all the reconstituted dyeings had side-to-side variations of less than two Starlab units.

Multi-shade dye series were conducted so that the shades dyed generally progressed from light to dark, although some dark to medium sequences were included. The resulting color differences were acceptable. Light-to-dark sequences are recommended for dyebath reuse, wherever possible. Drastic changes from a dark to a light shade should be avoided. The reason for this is as follows. Suppose the previous shade were dyed with a red dye at a concentration of 1 percent o.w.f. After dyeing, about 0.05 percent o.w.f. would remain, assuming 95 percent exhaustion. Assuming the accuracy of the dyebath analysis technique to be 10 percent, the amount of uncertainty in the remaining red dye concentration is 0.005 percent o.w.f. If the next shade to be dyed also had a red dye recipe amount of 1 percent o.w.f. or more, the relative uncertainty of the recycled dye amount is about 0.5 percent (0.005/1.0). However, if the next shade to be dyed is much lighter and requires only 0.1 percent o.w.f. red dye, then the relative error in the recycled dye amount is 5 percent (0.005/0.1). Large relative errors such as this should be avoided.

In summary, the results in Table VII-2 show a generally successful set of pilot-scale dyeings. As a result, preparations for full-scale dyebath reconstitution and reuse trials at Mill C-2 were started.

## ADVANTAGES AND LIMITATIONS OF PILOT-SCALE DYEINGS

Pilot-scale dyeings have a number of advantages, together with some disadvantages. These are discussed separately below.

### Advantages

The principal advantage of pilot-scale experiments is that they simulate full-scale mill dyeing much more closely than beaker dyeings, while still minimizing raw material requirements. This similarity allows additional variables and problems to be identified and resolved.

Pilot-scale experiments are more easily performed than full-scale reconstitution trials. These experiments do not require pumps, piping, flowmetering devices, or the development of complex logistics. In addition, experiments at this level do not interfere with production runs.

### Disadvantages

There are a number of limitations involved with pilot-scale reconstitution experiments, although most of them are minor. By its very nature, carpet committed to pilot-scale beck dyeings can be considered lost, i.e., it will not be sold. In addition, these trials require the full attention of at least one person. Without additional becks, and perhaps additional manpower, reconstitution series must be performed sequentially rather than simultaneously, a time-consuming task.

While pilot-scale dyeings simulate full-scale dyeings much more closely than do beaker dyeings, some aspects of pilot-scale dyeing will still be at variance. For example, in these pilot-scale beck dyeings, a single open-width carpet piece was dyed in each bath, whereas full-scale dyeings involve two carpets in rope form. In addition, certain quality control features cannot be evaluated with certainty with carpet dyed on such a small scale.

Although these laboratory reconstitution experiments are certainly very useful and advisable, the only way to prove the technical feasibility of implementing dyebath reconstitution and reuse at a mill is to perform full-scale trials at the mill on the basis of successful bench- and pilot-scale dyeings.

# SECTION VIII

# FULL-SCALE DYEBATH REUSE EXPERIMENTS

This section describes full-scale dyebath reconstitution and reuse trials performed at Mill C-2. It is organized as follows. The objectives of the full-scale demonstration are listed first. A number of preparatory activities are then reviewed that had to be carried out before reconstitution and reuse of dyebaths could occur. This is followed by a presentation of the procedures used to perform reuse dyeings at the mill. The full-scale dyeings are next described together with a discussion of the results. This section concludes with an evaluation of the prospects for implementation of this technology at Mill C-2.

## OBJECTIVES

The principal objective of full-scale reconstitution trials at the mill was to demonstrate this reuse method's technical feasibility. The mill demonstration would allow any problems that might develop at full-scale to be identified and resolved.

Another important objective of this effort was to collect water, chemical, and energy savings data associated with dyebath reconstitution. Environmental data on pollutant reductions due to dyebath reconstitution were not available elsewhere, and so these trials were also an opportunity to obtain these data. The environmental data developed during the full-scale reconstitution trials are presented separately in Section IX.

## PREPARATORY ACTIVITIES

### Demonstration Logistics

The first problem to be confronted in attempting to perform full-scale reconstitution dyeings at Mill C-2 was to determine the logistics of dyebath reuse. Three general approaches were considered:

1) reconstitute dyebath in-situ following hot-pull and separate rinsing of dyed carpet;

2) transfer exhausted dyebath to a holding tank, rinse and pull carpet in normal fashion, and return dyebath to beck for next dyeing;

3) transfer exhausted dyebath to an empty, adjacent dyebeck for reuse.

For the purposes of a one-week demonstration, the third approach was the simplest and most economical alternative.

Three adjacent becks with capacities of approximately 7,000 gallons each were selected for these trials. There were several important considerations in developing a system to transfer the exhausted dyebaths from one beck to another:

- the temperature of the dyebath to be transferred might be as high as 140°F;
- the dyebath transfer had to be accomplished relatively quickly; and
- the volume of dyebath transferred had to be known.

To satisfy these conditions, a dyebath transfer system, as described below, was developed.

### Dyebath Transfer Equipment

The heart of the dyebath transfer system was a 4-inch Peabody Barnes Model 4-STCCG centrifugal trash pump. This wheel-mounted portable pump was powered by a 30-horsepower Wisconsin 4-cycle gasoline engine and transferred approximately 500 gpm of exhausted dyebath.

The suction portion of the system consisted of a screen, piping, suction hose, and quick disconnect couplings. The purpose of the screen was to trap loose yarn and mats of fiber. It was fabricated of thin gage perforated (1/4" holes) stainless steel sheet with dimensions of 12" x 6" x 66" deep and was left open at the top. A lip was welded along a top edge of this elongated open box so that the screen could be slipped between the beck wall and an appropriately spaced railing each time an exhausted dyebath was to be transferred. The suction end of a 4" diameter x 68" long carbon steel pipe was inserted to within approximately 5" of the bottom of this box screen. An attached 4-inch, 90-degree elbow was chained to a support across the top of the beck. From the back of the beck this suction pipe/elbow was linked to the suction port of the pump by 25 feet of 4-inch spiral reinforced rubber suction hose; aluminum cam and groove fittings (quick disconnect couplings) at each end allowed this suction hose to be easily attached and removed from the suction pipe and the pump.

The discharge portion of the dyebath transfer system consisted of quick disconnect couplings, discharge hose, pipes, and a flowmeter. A 50-foot, 4-inch diameter mill hose was attached to the discharge of the pump using a quick disconnect coupling. The downstream end of this hose was connected to three feet of 4-inch carbon steel pipe by means of a similar fitting. This pipe led into a 4-inch Brooks Wafer-Mag electromagnetic flowmeter. A magnetic flowmeter was chosen for this application since loose fibers and yarns could interfere with the operation of positive displacement flowmeters. Cumulative gallons through the flowmeter were displayed on a resettable Brooks Model 410S Electronic Computing Totalizer. The 3-foot pipe, magnetic flowmeter, and totalizer display were mounted on a lightweight cart for portability. A second 50-foot mill hose was attached to the outlet side of the flowmeter with a quick-disconnect coupling. The other end of this hose was joined by similar means to a discharge piping arrangement consisting of 4-inch, 90-degree elbows on both ends of a 2-foot length of pipe. This arrangement was secured in place. The elbows were at right angles to each other to enable the exhausted dyebath to be discharged longitudinally along the front of the receiving beck.

Once all the dyebath transfer equipment had been assembled at the mill, several transfers were performed to "dry-run" the equipment and transfer procedures, and more importantly to calibrate the becks.

### Calibration of Becks

Accurate volume measurement is very important to successful reconstitution. Although use of a flowmeter would enable transfer volumes to be measured, it would not provide information on exhausted dyebath volumes in the beck prior or subsequent to pumping. For this study, it was desired to measure these volumes so that accurate data on water and energy savings could be obtained. For these reasons, and to verify flowmeter measurements, the dyebecks used in this demonstration were calibrated using the flowmeter prior to the start of the reconstitution experiments.

The baffle plates in the front of each beck consist of several regularly-spaced rows of one-inch diameter holes. These rows served as a good benchmark for calibrating the becks. The becks were calibrated in several ways.

Due to the irregular floor of the beck, and space taken up by carpet, volume measurements based on beck dimensions alone would have been difficult and subject to some uncertainty. However, by using the dyebath transfer equipment to fill an empty beck, flow meter readings could be used to determine the volume in the beck at each row. Flowmeter measurements between rows near the top of the beck were compared with estimated volumes based on beck dimensions and good correlations were observed. Subsequently, the becks were calibrated with Style 1 or Style 2 carpets in place in a similar manner using fresh water for transfer activities. Finally, the flowrate of cooling water into the becks was determined by using a stopwatch to determine how much time was required to reach various levels, i.e., known volumes, in the becks.

Once these various measurements were completed, specific procedures for exhausted dyebath transfer and other reconstitution activities could be developed.

## PROCEDURES

The typical dyeing procedure at Mill C-2 for these two carpet styles is as follows. Ranges are given to account for variations between becks and carpet styles.

Step 1. With beck drain closed, load two rolls of carpet into beck using a spray rinse to assist in moving carpet over the reel.

Step 2. Fill beck with cold water to approximately 5,600 to 5,800 gallons.

Step 3. Add auxiliary chemicals to the beck; continue mixing for five minutes after all chemicals have been added.

Step 4. Measure pH and adjust if necessary to attain 6.8 to 7.2 for Style 1 and 8.0 to 8.5 for Style 2.

Step 5. Add dyes and mix for ten minutes.

Step 6. Raise dyebath temperature to 180°F at 3°F per minute.

Step 7. Dye at 180°F for 30 minutes.

Step 8. Patch (i.e., inspect shade) and, if necessary to obtain acceptable dyeing, perform add(s). (After each add [chemical and/or dye addition], dyeing is performed for 15 additional minutes.)

Step 9. Add 4,500 gallons cold water with beck drain open to cool to 150°F or lower. Then shut water and drain beck.

Step 10. Close drain and fill beck with 6,300 to 6,500 gallons cold water to rinse, cool, and float carpet.

Step 11. Unload carpet.

Step 12. Drain and flush beck to prepare it for next dyeing.

Some procedural modifications, specifically in Steps 1, 2, and 9, were required for the reconstitution dyeings; Steps 3 through 8 and 10 through 12 remained unchanged. An outline of the reconstitution dyeing procedure is presented below.

Step 1. With beck drain open, load two rolls of carpet into beck using a spray rinse of about 800 gallons. (Tint that was added to the greige carpet is removed from carpet yarns during the spray rinse; for reconstituted dyeings, in contrast to conventional dyeings, the drain was left open to prevent tint buildup in the recycled dyebaths.)

Step 2. Close drain. Arrange and connect dyebath transfer equipment into appropriate orientation. Set flowmeter totalizer to zero. Pump 5,500 gallons of exhausted dyebath to new beck from beck used for previous dyeing.

| | |
|---|---|
| Steps 3. thru 8. | Same as conventional dyeing procedures for these steps. |
| Step 9. | Add cold water with beck drain closed and cool to approximately 140°F. (Some exhausted dyebath will be lost through overflow ports.) Collect dyebath sample for absorbance measurement. Pump to new beck (Step 2 of subsequent dyeing cycle). Perform solvent extraction of dyebath sample, perform absorbance measurements, and calculate amount of dyes and auxiliary chemicals required for next dyeing, using computer. Drain remaining volume from dyebeck. |
| Steps 10. thru 12. | Same as conventional dyeing procedure for these steps. |

The first dyeing in a reconstitution series was identical to a conventional dyeing through Step 8; the last dyeing in a series followed conventional dyeing procedures from Step 3 on.

As a precaution to prevent accidental draining of exhausted/reconstituted dyebaths, signs were posted on the appropriate dye beck and dye office control panel to indicate that there was an experimental dyeing in progress in that beck. In addition, constant supervision of the experimental dyeings was provided by project team members.

Auxiliary chemical make-up quantities were calculated by maintaining an estimated mass balance tally for each chemical in the dyebath and using this information to determine the make-up amounts necessary for the subsequent dyeing.

## RESULTS

### Series Description

Prior to initiation of full-scale trials, a ten-bath reconstitution series was planned for each of the two carpet styles. On the first day of the dyebath reuse trials, the production schedule for the entire week at the mill was reviewed. Ten shades of each carpet style were selected for dyeing using dyebath reconstitution. The shades were ordered in each series so that dyeing would progress from light to dark shades. In actuality, problems occurred in the third dyeing of the first series that caused this series to be aborted. A second series of Style 1 carpet was then started. Thus, three series of dyeings were actually conducted. A summary of these three series is presented in Table VIII-1. The formulations of the shades dyed in these series is presented in Table VIII-2. Notable features of the individual dyeings in each series are presented in Table VIII-3, including the add history of each dyeing, cooling water use, and dyebath temperature at the dyestuff add.

Series 24 was planned as a 10-bath trial. However, a yarn lot change in the Style 1 carpet after the first dyeing resulted in three adds being required to attain shade on the second dyeing. Even with three adds, a redye was required to attain the proper shade for this dyeing. Difficulties were also encountered in the third dyeing, with one of the two carpet rolls dyeing more heavily than the other. After an add, one of the carpets matched the standard shade while the other was still too light. With no beck available for transferring the exhausted dyebath into, it had to be dumped so that the successful carpet roll could be removed. Subsequently, a fresh bath was used to bring the second roll to standard. By dumping the bath, reconstitution series 24 ended.

Series 25 consisted of six dyeings of Style 1 carpet. No significant problems were encountered and all dyeings of this style were satisfactory.

Series 26 was a 10-bath reconstitution series using Style 2 carpet. As Table VIII-3 shows, with the exception of the first dyeing (a conventional dyeing) very few problems were encountered. Three of the last four dyeings in this series required no adds.

### Shade Matching

The carpet styles and shades used in these full-scale trials were selected at the beginning of the one-week demonstration from scheduled production for that period. With the exception of the procedural changes necessary to physically perform reconstitution, all prior and subsequent dyehouse activities relative to these dyeings were unchanged. They were treated as production runs by all mill personnel, and accordingly did not receive any more nor any less attention during the inspection process than conventionally dyed production. The success of

## TABLE VIII-1

## FULL-SCALE DYEING SERIES SUMMARY

| Series Number | Carpet Style | Number of Dyeings | Shade Dyeing Sequence |
|---|---|---|---|
| 24 | 1 | 3 | 111-111-101 |
| 25 | 1 | 6 | 101-112-112-103-113-113 |
| 26 | 2 | 10 | 211-211-212-212-213-214-215-215-216-217 |

## TABLE VIII-2

## FULL-SCALE DYEING SHADES AND FORMULATIONS

### Carpet Style 1

| | Dyestuffs (% owf) | | | | |
|---|---|---|---|---|---|
| | Shade 101 | Shade 103 | Shade 111 | Shade 112 | Shade 113 |
| **Dyestuff** | | | | | |
| Orange #1 | .04 | .05 | .045 | .06 | .09 |
| Red #1 | .02 | .05 | .009 | .03 | .14 |
| Blue #1 | .01 | .04 | .007 | .02 | .04 |

| Dyebath Auxiliary Chemicals (% owf) | |
|---|---|
| MSP | 1.0 |
| TSP | 1.0 |
| Sequestering Agent | 0.5 |
| Levelers | 2.1 |
| Defoamer | 0.85 |

### Carpet Style 2

| | Dyestuffs (% owf) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Shade 211 | Shade 212 | Shade 213 | Shade 214 | Shade 215 | Shade 216 | Shade 217 |
| **Dyestuff** | | | | | | | |
| Orange #1 | .011 | .02 | .030 | .03 | .06 | .07 | .11 |
| Red #1 | .002 | .01 | .009 | .02 | .03 | .04 | .13 |
| Blue #2 | .004 | .02 | .013 | .02 | .03 | .05 | 1.23 |

| Dyebath Auxiliary Chemicals (% owf) | |
|---|---|
| MSP | 0.3 |
| TSP | 0.85 |
| Sequestering Agent | 0.4 |
| Levelers | 1.6 |
| Defoamer | 0.65 |

## TABLE VIII-3
## FULL-SCALE DYEING DYEBATH SUMMARY

| | | | | | Add Summary * | | | Cooling Summary | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Series Number | Dyebath Number | Shade | Shade Description | Number of Adds | Add 1 | Add 2 | Add 3 | C.W. Gallons | Cooling End Temp °F | Initial Dyebath Temp °F | Remarks |
| 24 | 1 | 111 | Lt. Yellow | 0 | -- | -- | -- | 2,700 | 148 | 68 | -- |
| 24 | 2 | 111 | Lt. Yellow | 4 | D | D | D | 2,700 | 146 | 108 | a,b,c |
| 24 | 3 | 101 | Lt. Brown | 3 | D,B | D | D | -- | -- | 116 | c,d |
| 25 | 1 | 101 | Lt. Brown | 1 | D | -- | -- | 2,700 | 138 | 68 | |
| 25 | 2 | 112 | Lt. Golden Br. | 1 | D | -- | -- | 4,500 | 136 | 95 | |
| 25 | 3 | 112 | Lt. Golden Br. | 1 | D,L | -- | -- | 4,500 | 120 | 105 | |
| 25 | 4 | 103 | Med. Gray | 2 | D,L | D,L,B | -- | 4,500 | 138 | 105 | |
| 25 | 5 | 113 | Med. Plum | 2 | L | D | -- | 4,500 | 124 | 100 | |
| 25 | 6 | 113 | Med. Plum | 0 | -- | -- | -- | -- | -- | 105 | |
| 26 | 1 | 211 | Pale Yellow | 3 | L,B | B | D,L,B | 5,400 | 130 | 68 | c |
| 26 | 2 | 211 | Pale Yellow | 2 | D | D | -- | 3,600 | 120 | 105 | |
| 26 | 3 | 212 | Smoke | 1 | D,L | -- | -- | 4,500 | 133 | 92 | |
| 26 | 4 | 212 | Smoke | 1 | D,L | -- | -- | 2,700 | 137 | 102 | |
| 26 | 5 | 213 | Buff | 1 | D | -- | -- | 2,250 | 134 | 105 | |
| 26 | 6 | 214 | Beige | 2 | D | D | -- | 3,600 | 142 | 104 | e |
| 26 | 7 | 215 | Bronze | 0 | -- | -- | -- | 1,800 | 140 | 105 | |
| 26 | 8 | 215 | Bronze | 1 | L | -- | -- | 1,800 | 138 | 93 | |
| 26 | 9 | 216 | Tan | 0 | -- | -- | -- | 1,800 | 144 | 103 | |
| 26 | 10 | 217 | Royal Blue | 0 | -- | -- | -- | -- | -- | 116 | |

*Identifies addition of dyes (D) and/or leveler (L), and if add carried out at boil (B). Addition of defoamer is not noted.

Remarks

a - A fourth add consisting of addition of leveler and dyeing at the boil was also performed.
b - The two carpets removed from this dyebath after the fourth add subsequently needed to be redyed. These were the only redyes required out of 32 reconstituted dyeings (16 dyebaths).
c - Yarn lot change
d - Dyebath dropped after first add and one of two rolls removed; due to unlevel dyeing, remaining roll required two more adds until acceptable.
e - Dyeing accidently performed at boil; adds performed at 180°F.

these dyeings was to depend upon whether the various post-dyeing inspections resulted in their being shipped as first quality production without the need for redyes. No quantitative shade matching measurements were made. Based on these criteria, all dyeings were successful, and the redyes that occurred in series 24 were attributed to a yarn lot change rather than any problems caused by dyebath reuse.

### Water, Energy, and Chemical Savings

One of the incentives for dyebath reuse is the savings in water, energy, and auxiliary chemical usage. Their use was closely monitored during the reconstitution series. Table VIII-4 summarizes the savings that occurred due to dyebath reuse. Water and chemical savings range, for the most part, from 25 to 50 percent of normal usage. Dyestuff savings were minimal and are not listed on the table. Defoamer usage was the same in reuse dyeings as in conventional dyeings, i.e., all defoamer was assumed to have evaporated in a dye cycle. The results from series 24 have been omitted from the table, even though they also show comparable savings. As noted above, a yarn lot change in series 24 caused numerous problems with achieving the proper shade on the carpets and thus the series was aborted during the third dyeing. The cost savings associated with the data in Table VIII-4 are presented in Section X.

### Discussion

These full-scale experiments showed that the ease of obtaining satisfactory dyeings with reconstituted baths was comparable if not superior to that of conventional dyeing procedures. Both the number of adds and redyes were within the mill's typical frequency for these styles. The strict quality control standards of this mill were not an impediment to dyebath reuse.

The major problem encountered with dyebath reconstitution was that of pH control. Several pH adjustments of reconstituted dyebaths by MSP or TSP addition were required early in the dye series, especially with Style 1 carpet. However, as experience was gained with the behavior of these pH control agents and/or the recycled dyebath became more

## TABLE VIII-4

## WATER, ENERGY, AND CHEMICAL SAVINGS

| Dyeing | Water Savings (gallons) | Energy Savings (mm BTU) | Chemical Savings | | | |
|---|---|---|---|---|---|---|
| | | | M.S.P. (lb) | T.S.P. (lb) | Sequesterant (lb) | Leveler (gal.) |
| 25-1 | 2,400 | 0.0 | 0 | 0 | 0 | 0 |
| 25-2 | 5,000 | 1.33 | -11 | 19 | 5.5 | 3.0 |
| 25-3 | 5,200 | 1.83 | 9 | 14 | 5 | 1.5 |
| 25-4 | 4,000 | 1.83 | 4 | 14 | 5 | 1.5 |
| 25-5 | 3,600 | 1.58 | -1 | 14 | 5 | -0.5 |
| 25-6 | 5,300 | 1.83 | 9 | 14 | 5 | 1.5 |
| Totals | 25,500 | 8.40 | 10 | 75 | 25.5 | 7.0 |
| 26-1 | 100 | 0.0 | 0 | 0 | 0 | 0 |
| 26-2 | 4,700 | 1.78 | -1 | 11 | 5 | 2.5 |
| 26-3 | 4,700 | 1.15 | 6 | 12 | 5.5 | 2 |
| 26-4 | 7,000 | 1.63 | 0 | 13 | 5 | 1 |
| 26-5 | 7,650 | 1.78 | 3 | 11 | 6 | 2 |
| 26-6 | 6,300 | 1.73 | 3 | 11 | 6 | 2 |
| 26-7 | 6,500 | 1.78 | 2 | 12 | 5 | 1 |
| 26-8 | 6,500 | 1.20 | 2 | 14 | 6 | 0 |
| 26-9 | 8,100 | 1.68 | 4 | 14 | 6.5 | 3 |
| 26-10 | 7,900 | 2.30 | 4 | 15 | 6.5 | 2 |
| Totals | 59,450 | 15.03 | 23 | 113 | 51.5 | 15.5 |

buffered, no adjustments after addition of normal make-up auxiliary chemical quantities were required. Improved bath control may even be realized through reconstitution. This is because the effects of pH and chlorine variations in incoming source water are lessened resulting in more uniform dyebaths.

In spite of the success of these dyeings, there are some aspects of the reconstitution procedure used here that can be evaluated more fully. These aspects were not evaluated because the reuse procedures used in these trials were developed to emulate as closely as possible Mill C-2's conventional dyeing procedures. This approach was adopted to minimize the number of variables between conventional dyeings and reconstituted dyeings. However, these modifications and alternatives should be explored by the mill if dyebath reconstitution and reuse is implemented full-scale. In particular, the degree to which both cooling water volumes can be decreased and initial dyebath temperatures increased should be ascertained.

The purpose of the cool-down rinse is to cool the carpet to 150°F or lower before dropping the dyebath. The rinse water, however, also dilutes the exhausted dyebath, thereby requiring more auxiliary chemicals to be added to reconstitute the bath. Therefore, the savings achievable by dyebath reuse can be maximized by minimizing the amount of cool-down water used.

One alternative for reducing the amount of cool-down water is to use a bump-and-run dyeing procedure. The dyebath initially would be raised to 212°F (bump), held at the boil for 5 minutes, and then allowed to drift (run). The drift rate from the boil observed on several becks at Mill C-2 averages about 1.5°F/minute with beck doors closed. For a 30-minute dye cycle, 25 minutes of drifting would reduce the temperature to about 175°F. Further cooling during carpet sampling would reduce the temperatures to about 165°F, although the drift rate is much lower at these lower temperatures. With a starting temperature of 165°F before the cool-down rinse, the amount of water used for cooling could be reduced to about 1,500 gallons, compared to an average of 3,350 gallons used in these full-scale experiments.

A second alternative is to use the normal dye procedure, but keep the steam turned off after the carpet is sampled at the end of the dyeing process. This also would allow the dyebath temperature to drop to about 165°F before the start of the cool-down rinse.

In either case, the use of 1,500 gallons in the cool-down rinse would drop the dyebath temperature to about 145°F. The heat losses during pumping, storage, and holding in the new beck before the dyestuff add would further reduce the dyebath temperatures to approximately 110 to 115°F for the start of the next dyeing cycle. This starting temperature was found acceptable during the full-scale experiments. Even higher initial temperatures may be feasible, but this will have to be investigated. Several factors will influence this initial temperature decision including the rate of dye take-up by the fiber and the rate of dye leveling. For example, in this study it was observed that at temperatures near 100°F, the dye take-up rate of Style 1 carpet was very fast, whereas the rate for Style 2 carpet was slow.

## OUTLOOK FOR IMPLEMENTATION

The success of the dyeings performed in the reconstituted baths demonstrated the technical feasibility of dyebath reconstitution at this mill. The environmental and economic benefits of reconstitution are presented in the following two sections. Based on these results and estimates, Mill C-2 has made a firm decision to acquire, install, and modify necessary equipment in the near future to enable reconstitution to be implemented at certain becks.

# SECTION IX

# ENVIRONMENTAL BENEFITS OF DYEBATH REUSE

Batch dyeing is relatively inefficient in its use of auxiliary dyeing chemicals, water, and energy. At the end of a typical textile batch dyeing cycle, from hundreds to several thousand gallons of hot water containing most of the auxiliary chemicals initially added to the dyebath are discharged as wastewater. Reconstitution and reuse of these dyebaths rather than discharge may thus lead to several environmental benefits. Direct benefits are the reduction in wastewater volume and the expected reduction in pollutant discharge. Indirect benefits result from reductions in the consumption of chemicals and energy.

## OBJECTIVES

Several studies of dyebath reconstitution and reuse have been conducted in the past 8 years. These studies were concerned mostly with developing the methodology and quantifying the energy and chemical cost savings. Therefore, no data have been collected to date to quantify the potential environmental benefits of the technology.

It must be realized that, even with dyebath reuse, the actual quantities of water and pollutants discharged will vary from mill to mill. The following factors are among many that have been found to affect the discharge of wastewater from a dyehouse using dyebath reuse technology:

- Overflow or cool-down rinsing creates excess volumes of diluted dyebath that cannot be recycled.
- The number of dyeings made from the recycled dyebath will affect the discharge because the concentrations of pollutants in the dyebath generally increase with greater number of reuse cycles.
- Large quantities of steam condensate from live steam injection can result in excess volumes of water that cannot be recycled.

One of the objectives of this study was to characterize, at one mill, the actual wastewater discharge associated with dyebath reuse and to compare this discharge with that from conventional dyeing. The results at this one mill are considered to be indicative of the environmental benefits at other mills, though some mill-to-mill variation will be seen due to the factors noted above.

## METHODOLOGY

### Sampling and Preservation

All dyebath samples collected both during the pilot-scale and full-scale dyeings were grab samples. Before sampling, the contents of the dyebeck were allowed to mix well after any addition of chemicals, dyes, or water. Samples were collected from beneath the surface of the dyebath. Any large clumps of fiber or masses of yarn were removed from the samples to prevent them from adsorbing dyes or other constitutents of the dyebath during storage prior to analysis.

Temperature measurements were taken within the dyebeck at the time of sampling; pH measurements of the collected samples were taken in the field at this time also.

A one-liter aliquot of sample was acidified ($pH < 2$) with sulfuric acid and placed in a clean one-liter amber glass bottle for phenolics analysis. A one-liter aliquot of sample was placed in a clean plastic bottle with no preservative for analysis for alkalinity, BOD, suspended solids, and dissolved solids. A 0.5-liter aliquot was acidified ($pH < 2$) with sulfuric acid and placed in a clean plastic bottle for analysis for COD and total phosphorus. All samples were cooled to less than 4°C and packed in ice for overnight shipment to the laboratory.

### Sample Analysis

Sample analyses for BOD, COD, TSS, TDS, alkalinity, phenolics, and total phosphorus were conducted using EPA-approved methods as specified in FR44(233): 69464 to 69575 (December 3, 1979). Quality assurance procedures used are discussed in Appendix B.

Field measurements of pH were with a pH meter calibrated with standard buffer solutions (pH 4.01, 6.86, and 10.4). Temperature measurements were with a mercury thermometer (0 to 300°F) with calibration field-checked at 212°F (boiling water).

## PILOT-SCALE RESULTS

Wastewater data were collected during the pilot-scale dyeings for two reasons. First, it was desired to examine the potential buildup in concentration of various pollutants due to dyebath reuse. A large buildup could negate the reductions caused by recycle of the auxiliary chemicals. Second, it was desired to get actual measurements of the pollutant loadings resulting from dyebath reuse. Samples were taken of excess dye liquor that was discharged after each dyeing.

### Concentration Increase Due to Dyebath Reuse

Wastewater samples were taken during the various dye series to quantify the buildup of pollutants that occurs due to dyebath reuse. Table IX-1 presents the results of the sample analyses. The first column identifies the dye series and the dyeing within that series. For example, 17-1 identifies the first dyeing in reuse series 17; 17-5 identifies the fifth dyeing in that series. The samples were taken at the end of the dyeing process and represent the cooled, exhausted dyebath.

Note that the concentrations of BOD, COD, and dissolved solids all increase as the dyebath is reused. This is due to the buildup in impurities (e.g., yarn finishes) as the dyebath is recycled. The concentrations of suspended solids generally also increase, but there is much more variability in these data. The concentrations of phenolics are generally low and exhibit no discernible pattern. Total alkalinity and total phosphorus are measures of the buffering capacity of the dyebath (MSP and TSP buffering system) and remain relatively constant in each dyeing series. In some series, the phosphorus and alkalinity values actually decreased, indicating that the make-up quantities added to reconstitute the bath were somewhat lower than the quantities lost to the sewer. Even so, the buffering capacity of the dyebath was sufficient to maintain an acceptable pH during dyeing.

Figure IX-1 graphically shows the increase in pollutant concentrations caused by recycle. Data for COD and dissolved solids are plotted for each of the two carpet styles.

## TABLE IX-1

## PILOT-SCALE WASTEWATER DATA

Carpet Style 1

| Dye Cycle | $BOD_5$ | COD | TSS | TDS | Total Alkalinity* | Total Phenolics | Total Phosphorus |
|---|---|---|---|---|---|---|---|
| 17-1 | 1,040 | 2,340 | 43 | 1,260 | 143 | .060 | 112 |
| 17-2 | 1,270 | 3,250 | 6 | 1,390 | 139 | .082 | 112 |
| 17-3 | 1,630 | 4,090 | 11 | 1,620 | 139 | .100 | 116 |
| 17-4 | 1,870 | 4,760 | 41 | 1,680 | 122 | .087 | 124 |
| 17-5 | 2,240 | 5,810 | 45 | 1,880 | 139 | .114 | 120 |
| 18-1 | 970 | 2,260 | 18 | 1,320 | 135 | .049 | 140 |
| 18-5 | 2,240 | 5,640 | 106 | 1,720 | 122 | .059 | 93 |
| 19-1 | 870 | 2,300 | 6 | 1,180 | 129 | .073 | 106 |
| 19-2 | 1,550 | 3,730 | 11 | 1,430 | 141 | .082 | 99 |
| 19-4 | 1,970 | 5,440 | 40 | 1,730 | 139 | .140 | 86 |
| 19-5 | 2,060 | 6,070 | 124 | 1,750 | 133 | .092 | 79 |
| 19-10 | 3,140 | 6,670 | 95 | 2,280 | 131 | .074 | 86 |

Carpet Style 2

| Dye Cycle | $BOD_5$ | COD | TSS | TDS | Total Alkalinity* | Total Phenolics | Total Phosphorus |
|---|---|---|---|---|---|---|---|
| 20-1 | 653 | 2,780 | 4 | 1,350 | 228 | .042 | 102 |
| 20-6 | 2,340 | 6,430 | 10 | 2,300 | 139 | .066 | 56 |
| 21-1 | 970 | 2,740 | 5 | 1,230 | 143 | <.006 | 68 |
| 21-2 | 1,270 | 4,130 | 12 | 1,720 | 178 | .008 | 58 |
| 21-3 | 1,430 | 5,710 | 17 | 2,180 | 153 | <.006 | 69 |
| 21-4 | 1,900 | 7,100 | 19 | 2,750 | 149 | .072 | 60 |
| 21-5 | 1,940 | 7,580 | 22 | 2,900 | 137 | <.006 | 47 |
| 22-1 | 387 | 3,360 | 13 | 2,040 | 159 | .073 | 65 |
| 22-5 | 1,810 | 8,320 | 369 | 4,110 | 157 | .051 | 60 |
| 23-1 | 187 | 3,590 | 8 | 1,610 | 141 | <.006 | 61 |
| 23-2 | 1,020 | 4,140 | 10 | 2,130 | 135 | .060 | 55 |
| 23-5 | 768 | 8,280 | 276 | 3,910 | 151 | .065 | 42 |
| 23-6 | 1,760 | 7,180 | 133 | 3,280 | 114 | <.006 | 45 |
| 23-8 | 2,060 | 8,670 | 256 | 4,120 | 114 | .052 | 45 |
| 23-10 | 628 | 7,260 | 238 | 3,220 | 102 | .052 | 34 |

All units are mg/l.

*as $CaCO_3$

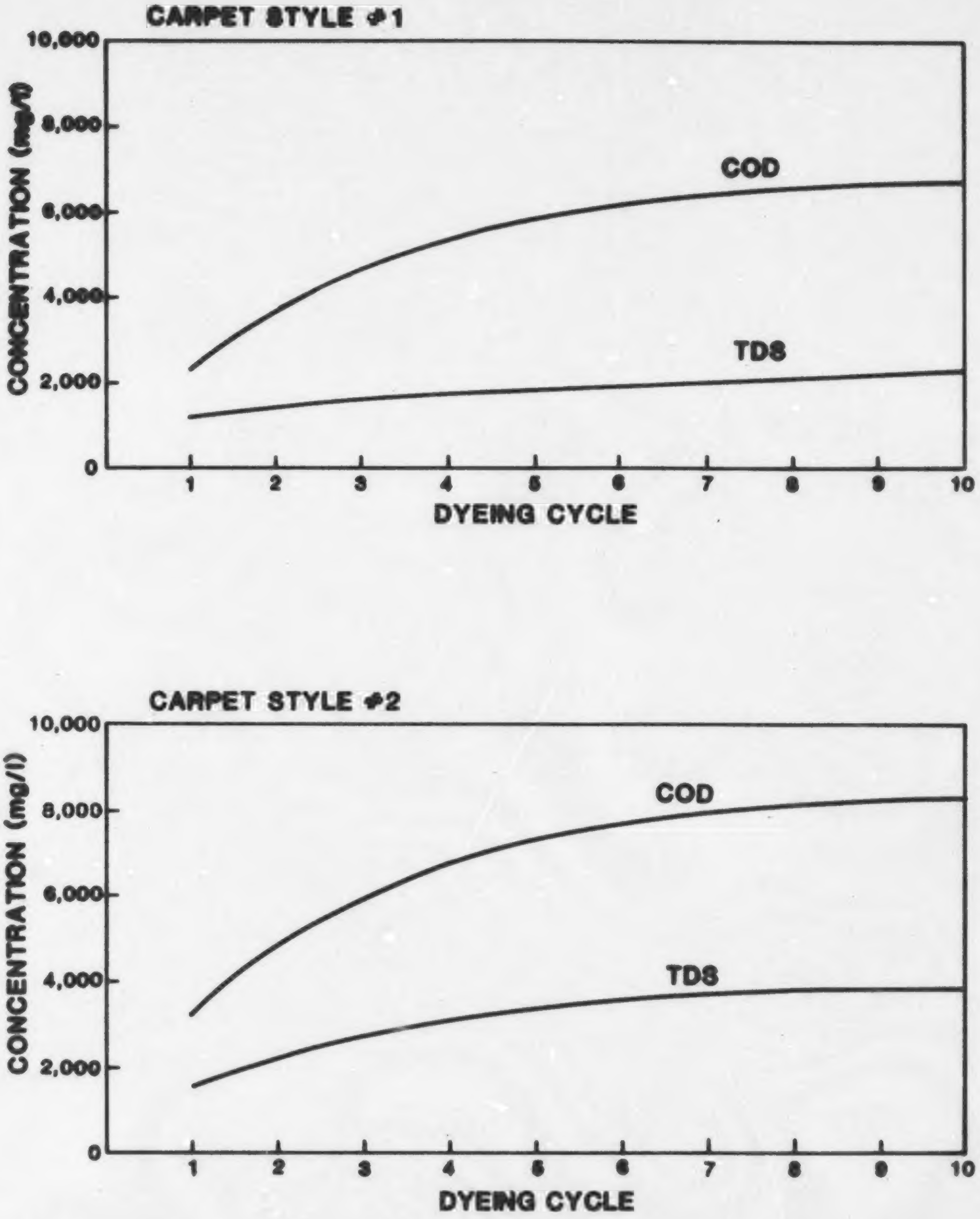

FIGURE IX-1
POLLUTANT CONCENTRATION VS DYEING CYCLE
PILOT-SCALE DATA

### Baseline

It was necessary to establish a baseline for the pollutant discharge from conventional dyeing. This was done by averaging the wastewater data collected for the first dyeing in each series. Pollutant loadings (mass of pollutant/mass of carpet) were calculated to account for the fact that the volume of wastewater and mass of carpet dyed varied from series to series. Table IX-2 presents the baseline pollutant loadings for conventional dyeing.

### Actual Discharge

Wastewater samples were collected after each dyeing in Series 17 (Style 1 carpet) and Series 21 (Style 2 carpet). The data from the analysis of these samples represent the discharge of pollutants from a series of 5 dyeings for each carpet style employing dyebath reuse. The actual discharge of pollutants was calculated for each dyeing, totaled, and converted to a pollutant loading. This loading was then compared to the baseline loading for conventional dyeing. In this fashion, the percent reduction in pollutant discharge due to dyebath reuse could be calculated. Table IX-3 presents the pollutant loadings for series 17 (Style 1 carpet) and the reduction in loading as compared to conventional dyeing. The same information is presented in Table IX-4 for series 21 (Style 2 carpet).

### Discussion

The concentrations of BOD, COD, TSS, and TDS all increased as the dyebaths were reused. By the fifth dyeing in each series, these concentrations were about double that of the first dyeing, or about double that of a conventional dyeing. Despite this increase, the total discharge of pollutants from a series of five dyeings using dyebath reuse was still substantially less than that from five conventional dyeings. The reduction in discharge of organics, as measured by BOD and COD, was about 30 to 35 percent. The reduction in dissolved solids was about 50 percent. The reduction in discharge of total-phosphorous was even greater, about 70 percent, due to the fact that total-phosphorus concentrations did not increase as the series of dyeings progressed.

## TABLE IX-2

## PILOT-SCALE POLLUTANT LOADING
## CONVENTIONAL DYEING

### Carpet Style 1

| Pollutant | Pollutant Loading for First Dyeing in Series | | | |
|---|---|---|---|---|
| | 17 | 18 | 19 | Average |
| BOD (g/kg) | 24 | 25 | 21 | 23 |
| COD (g/kg) | 54 | 58 | 56 | 56 |
| TSS (g/kg) | 1.0 | 0.5 | 0.1 | 0.5 |
| TDS (g/kg) | 29 | 34 | 29 | 31 |
| Phenolics (mg/kg) | 1.4 | 1.2 | 1.8 | 1.5 |
| Total-P (g/kg) | 2.5 | 3.5 | 2.5 | 2.8 |

### Carpet Style 2

| Pollutant | Pollutant Loading for First Dyeing in Series | | | | |
|---|---|---|---|---|---|
| | 20 | 21 | 22 | 23 | Average |
| BOD (g/kg) | 17 | 26 | -- | -- | 22 |
| COD (g/kg) | 73 | 74 | 82 | 93 | 80 |
| TSS (g/kg) | 0.1 | 0.1 | 0.3 | 0.2 | 0.2 |
| TDS (g/kg) | 35 | 33 | 50 | 42 | 40 |
| Phenolics (mg/kg) | 1.1 | 0.2 | 1.8 | 0.2 | 0.8 |
| Total-P (g/kg) | 2.6 | 1.8 | 1.6 | 1.5 | 1.9 |

## TABLE IX-3

## DYEBATH REUSE - POLLUTANT LOADING
## SERIES 17 - CARPET STYLE 1

| Pollutant | Pollutant Discharge for Dyeing Cycle | | | | | |
|---|---|---|---|---|---|---|
| | 17-1 | 17-2 | 17-3 | 17-4 | 17-5 | Total |
| BOD (g) | 8 | 38 | 50 | 17 | 237 | 350 |
| COD (g) | 19 | 98 | 127 | 43 | 616 | 903 |
| TSS (g) | 0.3 | 0.2 | 0.3 | 0.4 | 4.8 | 6.0 |
| TDS (g) | 10 | 42 | 50 | 15 | 200 | 317 |
| Phenolics (g) | 0.5 | 2.5 | 3.1 | 0.8 | 12 | 19 |
| Total-P (g) | 0.9 | 3.4 | 3.6 | 1.1 | 13 | 22 |

Total Carpet Mass Dyed in Series 17 = 23.0 kg

| Pollutant | Pollutant Loading | | |
|---|---|---|---|
| | Conventional | Dyebath Reuse | Reduction |
| BOD (g/kg) | 23 | 15 | 35% |
| COD (g/kg) | 56 | 39 | 30% |
| TSS (g/kg) | 0.53 | 0.26 | 51% |
| TDS (g/kg) | 31 | 14 | 55% |
| Phenolics (mg/kg) | 1.5 | 0.8 | 47% |
| Total-P (g/kg) | 2.8 | 1.0 | 64% |

## TABLE IX-4

## DYEBATH REUSE - POLLUTANT LOADING
## SERIES 21 - CARPET STYLE 2

| | Pollutant Discharge for Dyeing Cycle | | | | | |
|---|---|---|---|---|---|---|
| Pollutant | 21-1 | 21-2 | 21-3 | 21-4 | 21-5 | Total |
| BOD (g) | 45 | 25 | 16 | 72 | 233 | 391 |
| COD (g) | 127 | 83 | 66 | 270 | 910 | 1456 |
| TSS (g) | 0.2 | 0.2 | 0.2 | 0.7 | 2.6 | 3.9 |
| TDS (g) | 57 | 34 | 25 | 104 | 348 | 568 |
| Phenolics (mg) | 0.3 | 0.2 | 0.1 | 2.7 | 0.7 | 4.0 |
| Total-P (g) | 3.2 | 1.2 | 0.8 | 2.3 | 5.6 | 13 |

Total Carpet Mass Dyed in Series 21 = 27.2 kg

| | Pollutant Loading | | |
|---|---|---|---|
| Pollutant | Conventional | Dyebath Reuse | Reduction |
| BOD (g/kg) | 22 | 14 | 36% |
| COD (g/kg) | 80 | 54 | 32% |
| TSS (g/kg) | 0.2 | 0.14 | 30% |
| TDS (g/kg) | 40 | 21 | 48% |
| Phenolics (mg/kg) | 0.8 | 0.15 | 81% |
| Total-P (g/kg) | 1.9 | 0.5 | 74% |

One item to be noted is that these pilot-scale results may have slightly overestimated the reduction in pollutant discharge because the loadings from the final carpet rinse were not included. The method of rinsing used in the pilot-scale dyeings precluded the collection of wastewater data.

## FULL-SCALE RESULTS

Wastewater data were collected during the full-scale dyeings to confirm and refine the results of the pilot-scale dyeings. Wastewater samples were collected after each dyeing in series 25 (Style 1 carpet) and series 26 (Style 2 carpet). A sample of the dyebath immediately after dyeing was taken, as well as a sample of the cooled dyebath at the time of pumping. The final rinsewater for each dye cycle was also sampled.

### Concentration Increase Due to Dyebath Reuse

Table IX-5 presents the pollutant concentration data in the dyebath following each dyeing. These data also show a general increase in concentration as found during the pilot-scale dyeings. Increases in the concentrations of BOD, COD, TSS, and TDS were noted, but the amount of increase was less than in the pilot-scale dyeings. This could be due to the greater dilution caused by cooling water in the full-scale dyeings. Again, the concentrations of total phosphorus and alkalinity remained reasonably constant, a result of maintaining the MSP-TSP buffering system. The phenolics data were again variable with no apparent pattern.

### Baseline

Baseline loadings for full-scale conventional dyeing are summarized in Table IX-6 for each carpet style, based on the average of several samples. The pilot-scale loadings are also shown for comparison purposes. In general, the full-scale loadings are higher than those found during pilot-scale testing. This is, in part, because the full-scale data include loadings from rinse-water discharge. In addition, a slightly different formulation of auxiliary chemicals was being used in

## TABLE IX-5

## FULL-SCALE WASTEWATER DATA

Carpet Style 1

| Dye Cycle | $BOD_5$ | COD | TSS | TDS | Total Alkalinity* | Total Phenolics | Total Phosphorus |
|---|---|---|---|---|---|---|---|
| 25-1 | 435 | 2,590 | 16 | 1,220 | 187 | .052 | 122 |
| 25-2 | 735 | 3,770 | 26 | 1,730 | 188 | <.006 | 198 |
| 25-3 | 540 | 3,270 | 22 | 1,540 | 186 | .378 | 171 |
| 25-4 | 645 | 3,730 | 16 | 1,590 | 177 | .297 | 148 |
| 25-5 | 600 | 3,130 | 4 | 1,650 | 174 | .076 | 171 |
| 25-6 | 630 | 2,950 | 12 | 1,410 | 172 | .286 | 106 |

Carpet Style 2

| Dye Cycle | $BOD_5$ | COD | TSS | TDS | Total Alkalinity* | Total Phenolics | Total Phosphorus |
|---|---|---|---|---|---|---|---|
| 26-1 | 1,700 | 4,480 | 6 | 2,070 | 214 | .094 | 87 |
| 26-2 | 1,700 | 4,440 | 2 | 1,710 | 234 | <.006 | 101 |
| 26-3 | 1,650 | 4,890 | 21 | 1,830 | 230 | .194 | 77 |
| 26-4 | 2,055 | 5,210 | 18 | 1,880 | 243 | .009 | 108 |
| 26-5 | 1,830 | 5,150 | 30 | 1,990 | 240 | <.006 | 96 |
| 26-6 | 2,180 | 5,000 | 53 | 2,090 | 244 | .455 | 89 |
| 26-7 | 2,670 | 5,700 | 49 | 2,150 | 249 | <.006 | 99 |
| 26-8 | 2,610 | 7,240 | 130 | 2,620 | 277 | .144 | 106 |
| 26-9 | 2,550 | 7,300 | 25 | 2,630 | 262 | .376 | 101 |
| 26-10 | 2,760 | 7,310 | 77 | 2,500 | 263 | .241 | 95 |

All units are mg/l.

*as $CaCO_3$

## TABLE IX-6

## FULL-SCALE POLLUTANT LOADING CONVENTIONAL DYEING

Carpet Style 1

| Pollutant | Full-Scale Loading | Pilot-Scale Loading |
|---|---|---|
| BOD (g/kg) | 15 | 23 |
| COD (g/kg) | 92 | 56 |
| TSS (g/kg) | 0.77 | 0.5 |
| TDS (g/kg) | 45 | 31 |
| Phenolics (mg/kg) | 2.9 | 1.5 |
| Total - P (g/kg) | 3.6 | 2.8 |

Carpet Style 2

| Pollutant | Full-Scale Loading | Pilot-Scale Loading |
|---|---|---|
| BOD (g/kg) | 36 | 22 |
| COD (g/kg) | 96 | 80 |
| TSS (g/kg) | 0.23 | 0.2 |
| TDS (g/kg) | 49 | 40 |
| Phenolics (mg/kg) | 2.5 | 0.8 |
| Total - P (g/kg) | 1.8 | 1.9 |

the mill at the time of the full-scale dyeings, as compared to the formulation used in the pilot-scale dyeings.

## Actual Discharge

The volume of wastewater and quantities of pollutants discharged were then calculated for each dye cycle, based on the results of the sample analyses. The modified procedure for dyebath reuse included a pre-rinse to remove tinting material from the carpet before dyeing. The volume of wastewater and quantities of pollutants from the pre-rinse discharge were calculated and added to the above discharges for each cycle.

Table IX-7 summarizes the wastewater discharge for the six dyeings in series 25 (Style 1 carpet). These data are then totaled, loadings calculated based on the weight of carpet dyed in the series, and these loadings compared to the baseline loadings to compute the reduction in discharge due to dyebath reuse. Table IX-8 presents similar data for the ten dyeings in series 26 (Style 2 carpet).

## Effect of Series Length

The full-scale sampling data from series 26 (ten dyeings) was analyzed to determine the effect of series length, i.e., the number of dyeings conducted with the dyebath before dumping, on the reduction in pollutant discharge. Figure IX-2 graphically shows the reduction of COD and TDS as a function of series length. The data suggest that a series length of at least five dyeings is necessary to maximize the environmental benefits of dyebath reuse.

## Discussion

The full-scale sampling confirmed the increase in pollutant concentrations in the dyebath due to reuse. It also confirmed that despite this increase, a substantial reduction in the amount of pollutants discharged is achievable with dyebath reuse. In both full-scale series, the reduction in organics discharge was about 33 percent, although the BOD data for series 25 showed only a 13 percent reduction. Dissolved solids discharges were reduced from 25 percent with Style 1

## TABLE IX-7

## ACTUAL WASTEWATER DISCHARGE
## SERIES 25 - CARPET STYLE 1

|  | Volume | Pollutant Discharge (lbs) |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| Dyeing | (gal) | BOD | COD | TSS | TDS | Phenolics | Total-P |
| Conventional* | 17,400 | 29 | 180 | 1.5 | 88 | .006 | 7.1 |
| 25-1 | 9,500 | 15 | 72 | 0.2 | 40 | .004 | 3.4 |
| 25-2 | 12,600 | 33 | 132 | 1.2 | 63 | <.001 | 5.9 |
| 25-3 | 12,200 | 16 | 122 | 1.2 | 63 | .022 | 5.7 |
| 25-4 | 13,400 | 23 | 131 | 0.8 | 66 | .010 | 4.7 |
| 25-5 | 13,500 | 23 | 93 | 0.3 | 62 | <.001 | 4.9 |
| 25-6 | 18,200 | 41 | 194 | 1.0 | 103 | .019 | 6.7 |
| Total | 79,400 | 151 | 744 | 4.7 | 397 | .057 | 31.3 |

|  | Pollutant Loading |  |  |
|---|---|---|---|
| Pollutant | Conventional | Dyebath Reuse | Reduction |
| Flow (gal/lb) | 8.87 | 6.75 | 24% |
| BOD (g/kg)** | 15 | 13 | 13% |
| COD (g/kg) | 92 | 63 | 32% |
| TSS (g/kg) | 0.77 | 0.40 | 48% |
| TDS (g/kg) | 45 | 34 | 25% |
| Phenolics (mg/kg) | 2.9 | 4.8 | 0% |
| Total-P (g/kg) | 3.6 | 2.6 | 28% |

* For comparison to actual discharges.

** Equivalent to lb/1000 lb.

## TABLE IX-8

## ACTUAL WASTEWATER DISCHARGE
## SERIES 26 - CARPET STYLE 2

| | | Pollutant Discharge (lbs) | | | | | |
|---|---|---|---|---|---|---|---|
| Dyeing | Volume (gal) | BOD | COD | TSS | TDS | Phenolics | Total-P |
| Conventional* | 17,100 | 94 | 250 | 0.6 | 127 | .006 | 4.7 |
| 26-1 | 11,500 | 51 | 141 | 0.1 | 81 | .005 | 2.8 |
| 26-2 | 12,100 | 48 | 130 | <0.1 | 58 | <.001 | 3.2 |
| 26-3 | 12,800 | 38 | 147 | 1.3 | 67 | .007 | 2.5 |
| 26-4 | 10,000 | 55 | 127 | 0.7 | 50 | .001 | 2.4 |
| 26-5 | 9,150 | 33 | 114 | 1.5 | 51 | .001 | 1.8 |
| 26-6 | 11,400 | 67 | 153 | 3.3 | 77 | .022 | 3.2 |
| 26-7 | 11,200 | 63 | 145 | 2.3 | 55 | <.001 | 2.0 |
| 26-8 | 10,700 | 61 | 176 | 5.4 | 72 | .005 | 2.1 |
| 26-9 | 8,700 | 59 | 116 | 0.8 | 61 | .018 | 1.7 |
| 26-10 | 14,600 | 156 | 416 | 4.5 | 151 | .015 | 5.3 |
| Total | 112,150 | 631 | 1,665 | 20.0 | 723 | .076 | 27.0 |

| | Pollutant Loading | | |
|---|---|---|---|
| Pollutant | Conventional | Dyebath Reuse | Reduction |
| Flow (gal/lb) | 6.58 | 4.31 | 34% |
| BOD (g/kg)** | 36 | 24 | 33% |
| COD (g/kg) | 96 | 64 | 33% |
| TSS (g/kg) | 0.23 | 0.78 | 0% |
| TDS (g/kg) | 49 | 28 | 43% |
| Phenolics (mg/kg) | 2.5 | 2.9 | 0% |
| Total-P (g/kg) | 1.8 | 1.0 | 44% |

* For comparison to actual discharges.

** Equivalent to lb/1000 lb.

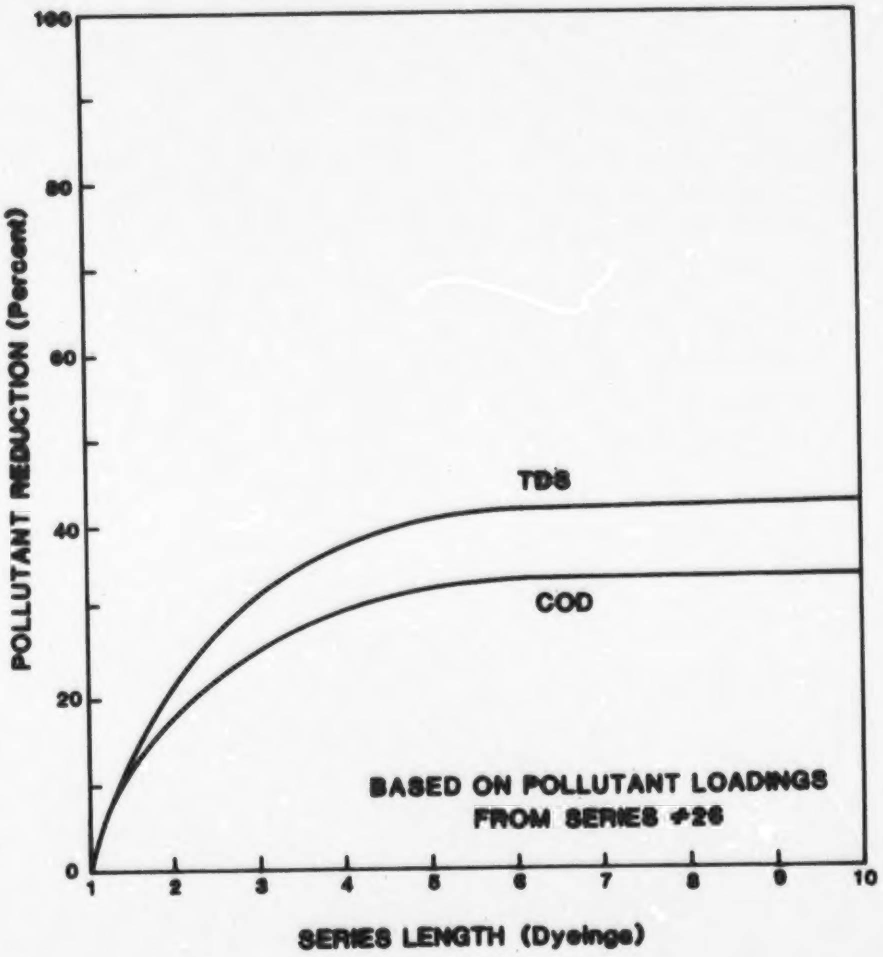

FIGURE IX-2
EFFECT OF SERIES LENGTH
ON POLLUTANT REDUCTION

carpet to 43 percent with Style 2 carpet. Reductions in total-phosphorus discharge were very similar to the dissolved solids results. Wastewater flow was also reduced: 24 percent with Style 1, and 34 percent with Style 2.

Compared to the pilot-scale results, the reductions found during the full-scale dyeings are generally smaller. In part this is due to the fact that the full-scale data include rinsewater discharges whereas the pilot-scale data did not. More important is the fact that more cooling water was used during the full-scale dyeings. This led to more dilution of the exhausted dyebath and consequently less recycle (in a given volume of dyebath) of auxiliary chemicals. Thus, losses to the sewer were greater. This suggests that decreasing the amount of cooling water used will increase the environmental benefits of dyebath reuse. Methods for reducing the amount of cooling water needed were discussed in Section VIII.

One final observation is the unexplained buildup of suspended solids in series 26 (Style 2 carpet). This buildup was more dramatic than for other pollutants and resulted in the discharge of more suspended solids during the series of ten dyeings employing dyebath reuse than in ten conventional dyeings. The total discharge of suspended solids in series 26 was about 20 pounds in 112,000 gallons, or about 21 mg/l. A buildup of this magnitude was not noted in series 25 (Style 1 carpet).

# SECTION X

# ECONOMIC ANALYSIS

The full-scale demonstration of dyebath reconstitution and reuse at Mill C-2 showed that significant savings in the usage of chemicals, water, and energy are possible by dyebath reuse. The environmental benefits from these raw material savings are also substantial. However, this recycle technology must have favorable economics if it is to be widely implemented in the textile industry.

This section of the report examines the cost-effectiveness of dyebath reconstitution as a recycle technology. The economic analysis is based on the results of the full-scale demonstration. Cost savings, capital costs, and yearly operating/maintenance costs are developed. A simple payback period analysis and a return-on-investment analysis are then presented.

It should be emphasized that Mill C-2 is not an ideal or best-case candidate for dyebath reuse. The relatively high liquor ratio used in dyeing, and a low auxiliary chemical value (about 3 cents per pound of fiber) both have adverse effects on the economics of reconstitution. In addition, the use of a cool-down or overflow rinse further decreases the savings obtained. Nevertheless, the payback period for dyebath reuse at this mill, as shown below, is still less than 2 years, which is generally considered to be acceptable in the industry. The possibility of reducing this payback period further by optimizing the dyeing procedure for dyebath reuse is examined at the end of this section.

## COST SAVINGS

The quantities of dyes, auxiliary chemicals, water, and energy saved through dyebath reuse during the full-scale demonstration of dyebath reconstitution are tabulated in Section VIII. The dollar savings associated with these items are presented in Tables X-1 and X-2 for series 25 (Style 1 carpet) and series 26 (Style 2 carpet) respectively. The results of series 24 have been omitted since this series consisted of only three dyeings.

## TABLE X-1

## FULL-SCALE DYEBATH REUSE COST SAVINGS

Series 25 (Carpet Style 1 : 6 dyeings)

| | Savings from Reuse | | Percent Savings |
|---|---|---|---|
| Cost Item | Quantity | Dollars | vs. Normal Dyeing |
| Dyes - Orange | 0.034 lb | $0.10 | 1 |
| Red | 0.061 lb | 0.50 | |
| Blue | 0.025 lb | 0.25 | |
| Chemicals - MSP | 10 lb | 5.50 | 27 |
| TSP | 75 lb | 24.30 | |
| Sequesterant | 25.5 lb | 10.70 | |
| Leveler | 7 gal | 51.80 | |
| Water/Sewer | 25,500 gal | 20.15 | 26 |
| Energy (Steam) | 10,900 lb | 29.80 | Not estimated |
| | Total | $143.10 | |

Average per cycle savings = $23.85 (based on 6 cycles)

## TABLE X-2

## FULL-SCALE DYEBATH REUSE
## COST SAVINGS

Series 26 (Carpet Style 2 : 10 dyeings)

| Cost Item | Savings from Dyeing<br>Quantity | <br>Dollars | Percent Savings<br>vs. Normal Dyeing |
|---|---|---|---|
| Dyes - Orange | 0.044 lb | $0.10 | 1 |
| Red | 0.015 lb | 0.10 | |
| Blue | 0.014 lb | 0.10 | |
| Chemicals - MSP | 23 lb | 12.80 | 35 |
| TSP | 113 lb | 36.70 | |
| Sequesterant | 51.5 lb | 21.60 | |
| Leveler | 15.5 gal | 114.70 | |
| Water/Sewer | 59,500 gal | 47.00 | 36 |
| Energy (Steam) | 19,500 lb | 53.30 | Not estimated |
| | Total | $286.40 | |

Average per cycle savings = $28.60 (based on 10 cycles)

Note that for both series, the savings in auxiliary chemical costs account for the largest portion of the total savings, approximately 65 percent. Energy savings account for about 20 percent of the total savings. Water and sewer savings account for the balance, about 15 percent. Dye savings in both series were very small. Dye savings are largely dependent on the shades being dyed. Deeper shades will have larger quantities of dyestuffs remaining to be recycled with the exhausted dyebath. Series 25 and 26 consisted of mainly light and medium shades, and the dyestuff exhaustions were very high, typically greater than 98 percent, with these lighter shades. Thus, only small quantities of dyestuffs remained in the exhausted dyebaths for recycle.

The average savings per dye cycle was $23.85 for Style 1 carpet and $28.60 for Style 2 carpet. Most of this difference is due to the fact that less water (about 1,300 gallons per cycle less) was used in the cooldown rinse for series 26 (Style 2). This resulted in less dilution and therefore greater savings in auxiliary chemicals. As noted on Tables X-1 and X-2, Series 25 had a 27 percent savings in auxiliary chemical costs as compared to conventional dyeing. The smaller dilution in series 26 increased this savings to 35 percent, thus resulting in a greater dollar savings per cycle.

## CAPITAL COST

In order to evaluate the cost-effectiveness of dyebath reconstitution as a recycle technology, the capital cost for implementing it at Mill C-2 was estimated. For an initial design, it was assumed that two dyebecks at the mill would be dedicated to dyebath reconstitution. These two becks would be served by a simple pump and holding tank.

Following the dye cycle, the exhausted dyebath is pumped to an elevated holding tank. The carpet is then rinsed in the beck and pulled in the normal fashion. A new greige carpet is then loaded into the now-empty beck and the dyebath returned for reuse from the holding tank by gravity.

## Cost for Pumping System

Figure X-1 depicts a flow schematic for the dyebath reuse pumping system. Figure X-2 is an isometric piping diagram that shows the general configuration and dimensions used in estimating the capital cost. Specifications for mechanical equipment and piping needed for the dyebath pumping system are listed in Table X-3. These specifications are for cost estimating purposes only and are not intended to be final design criteria.

Pages 98-101 present the construction cost estimate for the dyebath reuse pumping system described above. The estimate assumes that an outside mechanical contractor is hired to perform the work. The mechanical contractor hires an electrical subcontractor for electrical work. Material prices are for October 1983; labor rates are $18 per hour for mechanical work and $19.50 per hour for electrical work. Other assumptions include 4 percent sales tax on all materials and equipment, 10 percent labor overhead, 15 percent contractor profit, 10 percent markup on electrical work by mechanical contractor, and an allowance of about 10 percent for general conditions. Note that substantial cost savings would be achieved (on the order of 20 percent of total capital cost) if major portions of the work can be completed with in-house personnel.

## Analytical Equipment and Developmental Costs

Equipment required for dyebath analysis and makeup quantity calculations includes a spectrophotometer, computer, interface, and laboratory glassware. Expanding the dyebath reconstitution system in the future would require only additional pumps, piping, and tanks. No increase in the analytical equipment is required.

Table X-4 summarizes the capital cost for necessary analytical equipment, glassware, and miscellaneous supplies. In addition to these costs, an allowance of $10,000 is added to the capital cost to include the labor required to collect necessary data and conduct test dyeings before implementing dyebath reuse full-scale.

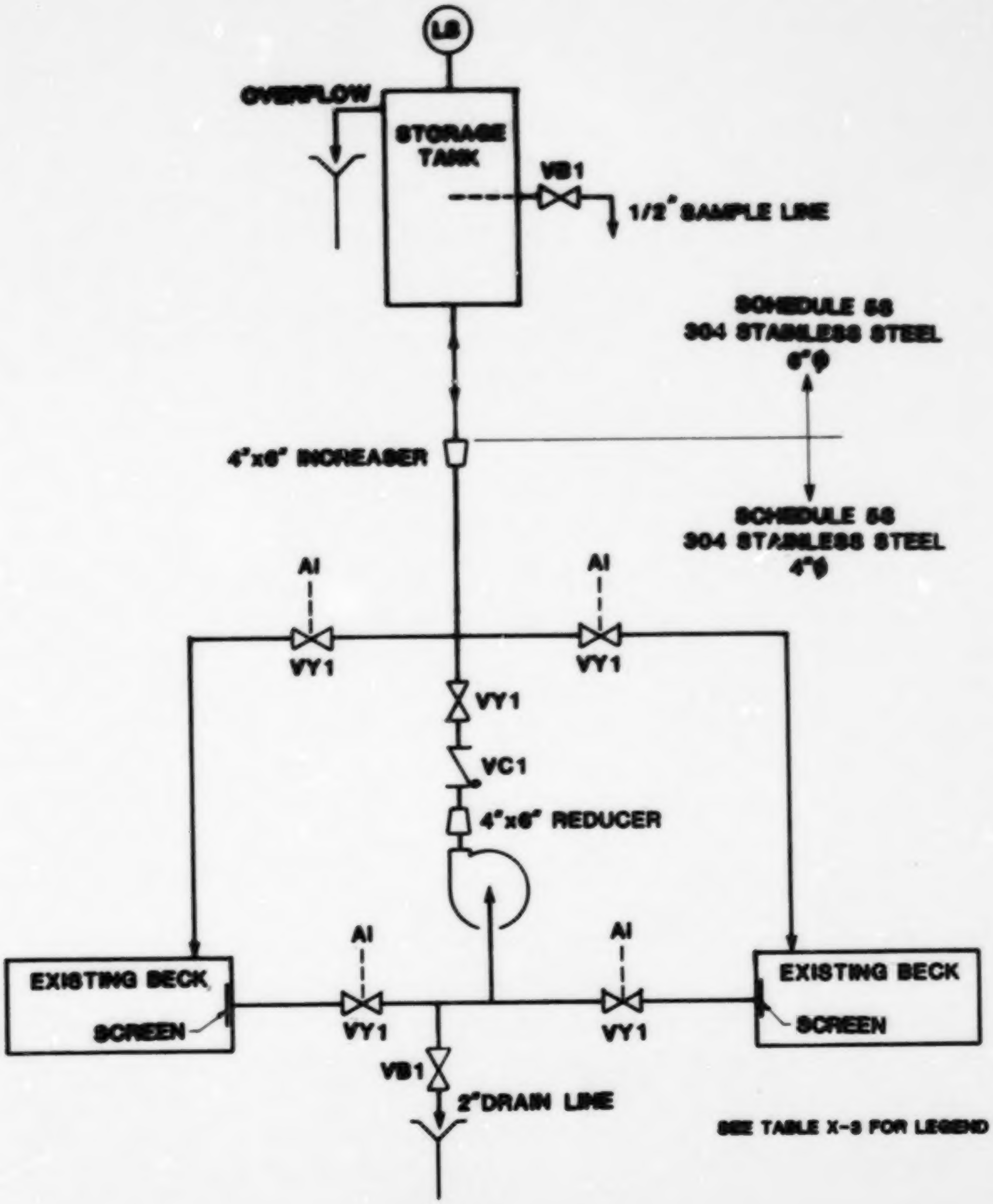
LS
OVERFLOW
STORAGE TANK
VB1
1/2" SAMPLE LINE
SCHEDULE 5S
304 STAINLESS STEEL
6"φ
4"x6" INCREASER
SCHEDULE 5S
304 STAINLESS STEEL
4"φ
AI
VY1
AI
VY1
VY1
VC1
4"x6" REDUCER
AI
AI
EXISTING BECK
SCREEN
VY1
VY1
EXISTING BECK
SCREEN
VB1
2"DRAIN LINE
SEE TABLE X-3 FOR LEGEND

FIGURE X-1
FLOW SCHEMATIC
TWO BECK RECONSTITUTION SYSTEM

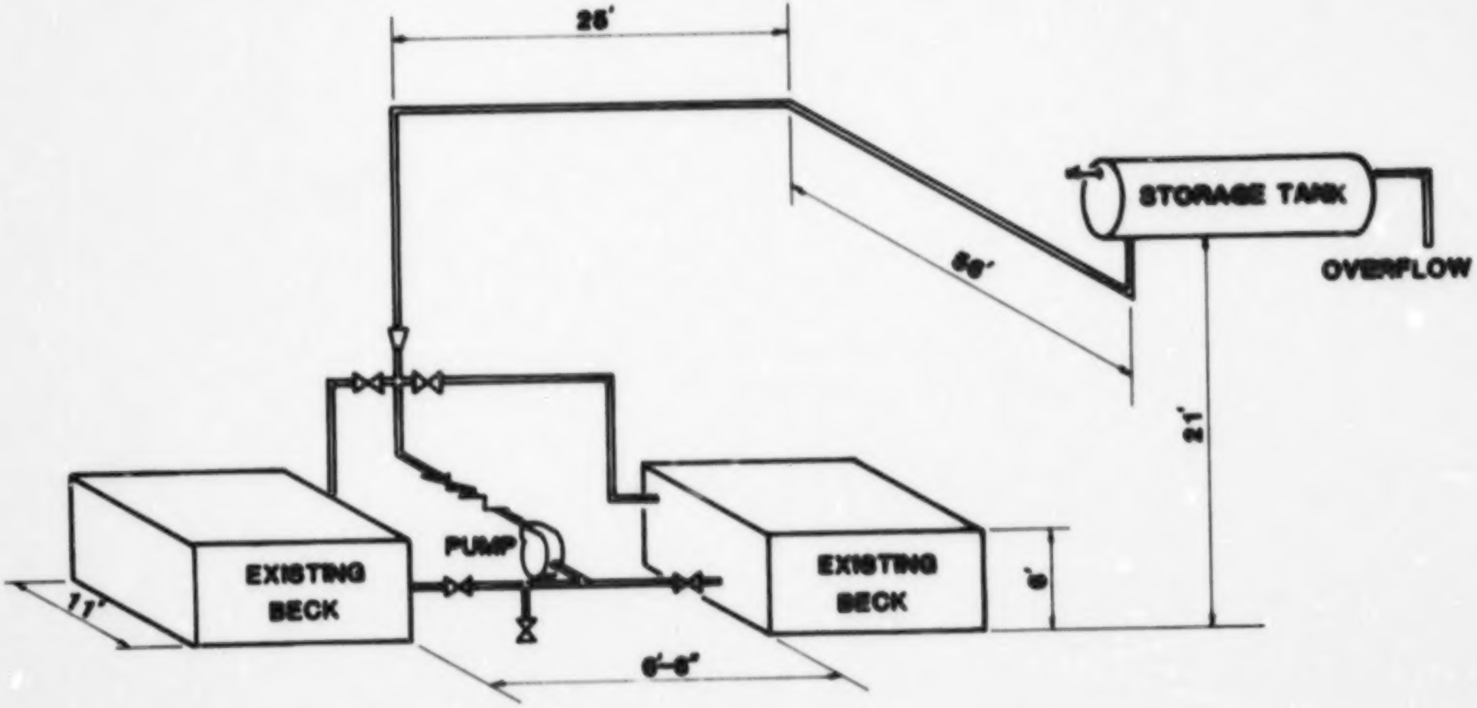
28'
58'
STORAGE TANK
OVERFLOW
21'
PUMP
EXISTING BECK
EXISTING BECK
11'
6'-6"
6'

FIGURE X-2        PRELIMINARY PIPING DESIGN

TABLE X-3

FULL-SCALE DESIGN DATA

| | | |
|---|---|---|
| Pump: | 316 stainless steel, centrifugal 4" x 6" pump with 1750 rpm, 15 hp, 460 volt electric motor. | |
| Tank: | 6,000 gallon, horizontal, insulated glass fiber-reinforced plastic (FRP) tank with manway, flanged inlet/outlet, overflow, and sampling tap. | |
| Piping: | Type 304 stainless steel (ASTM A-312), schedule 5S with welded joints and flanged connections. | |
| Valves: | VY1 - | 4-inch iron butterfly valve with full inside neoprene seat/lining, 316 stainless steel disc, pneumatic actuator, wafer type. |
| | VC1 - | 4-inch, 316 stainless steel swing check valve with flanged ends. |
| | VB1 - | 1/2- to 2-inch type 316 stainless steel ball valve with teflon seat and seal. |
| Level Switch: (LS) | Float-type level switch to turn off pump when 5500 gallon level is reached in the holding tank. | |
| Controls: | Pumps and valves controlled by signals from existing beck controllers, with local override. | |

# SVERDRUP & PARCEL
# CONSTRUCTION COST ESTIMATE

PAGE ______
SHEET 1 SHEETS THIS SET 4

JOB NO. 7518 CLIENT EPA-IERL

PROJECT Dyebath Reuse

PHASE OR DIVISION Project Cost Summary

BASIS FOR ESTIMATE
☐ NO DESIGN COMPLETED
☒ PRELIMINARY DESIGN
☐ FINAL DESIGN
☐

OFFICE St. Louis SECTION Env. BY JFB CHKD SAS DATE 10/10/83

| DESCRIPTION | QUANTITY |  | MATERIAL OR EQUIPMENT |  | LABOR OR LUMP SUM OR SUBCONTRACT |  | TOTAL COST |
|---|---|---|---|---|---|---|---|
|  | NO. UNITS | UNIT MEAS. | PER UNIT | COST | PER UNIT | COST |  |
| Equipment and piping |  |  |  | 32 802 |  | 8 089 | 40 891 |
| Instrumentation |  |  |  | 2 775 |  | 1 207 | 3 982 |
| Subtotal |  |  |  | 35 577 |  | 9 296 | 44 873 |
| Sales Tax (4%) |  |  |  | 1 423 |  | --- | 1 423 |
| Subtotal |  |  |  | 37 000 |  | 9 296 | 46 296 |
| Labor Overhead (10%) |  |  |  | --- |  | 930 | 930 |
| Subtotal |  |  |  | 37 000 |  | 10 226 | 47 226 |
| Profit (15%) |  |  |  | 5 550 |  | 1 534 | 7 084 |
| Subtotal |  |  |  | 42 550 |  | 11 760 | 54 310 |
| Electrical Subcontract |  |  |  | 2 133 |  | 1 628 | 3 761 |
| General Conditions |  |  |  | 4 000 |  | 1 000 | 5 000 |
| TOTAL |  |  |  | 48 683 |  | 14 388 | 63 071 |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  | Say | $63 000 |

## SVERDRUP & PARCEL
## CONSTRUCTION COST ESTIMATE

PAGE ______

SHEET 2 SHEETS THIS SET 4

JOB NO. 7518 CLIENT EPA-IERL

PROJECT Dyebath Reuse

PHASE OR DIVISION Mechanical Equipment and Piping

OFFICE St. Louis SECTION Cost Control BY MFF CHKD JDU DATE 10/10/83

BASIS FOR ESTIMATE
☐ NO DESIGN COMPLETED
☒ PRELIMINARY DESIGN
☐ FINAL DESIGN
☐

| | DESCRIPTION | QUANTITY | | MATERIAL OR EQUIPMENT | | LABOR OR LUMP SUM OR SUBCONTRACT | | TOTAL COST |
|---|---|---|---|---|---|---|---|---|
| | | NO. UNITS | UNIT MEAS. | PER UNIT | COST | PER UNIT | COST | |
| 1 | 6000 gallon FRP tank | 1 | EA | | 11 000 | | 828 | 11 828 |
| 2 | Tank saddles and supports | 2 | EA | | 400 | | 331 | 731 |
| 3 | 4 x 6 Pump 15 hp | 1 | EA | | 4 600 | | 497 | 5 097 |
| 4 | Pump Pad | 1 | EA | | 150 | | 124 | 274 |
| 5 | Hoisting and Rigging | 1 | Lot | | 1 000 | | 497 | 1 497 |
| 6 | 2-ft. sq. Screen and Frame | 2 | EA | | 200 | | 166 | 366 |
| 7 | 4" flanged nozzles @ becks | 4 | EA | | 640 | | 662 | 1 302 |
| 8 | 2" pipe, Sch 5S, 304 | 15 | LF | | 78 | | 41 | 119 |
| 9 | 4" pipe, Sch 5S, 304 | 30 | LF | | 390 | | 124 | 514 |
| 10 | 6" pipe, Sch 5S, 304 | 110 | LF | | 2 640 | | 615 | 3 255 |
| 11 | 2" 90° Elbow | 2 | EA | | 19 | | 108 | 127 |
| 12 | 4" 90° Elbow | 5 | EA | | 190 | | 435 | 625 |
| 13 | 6" 90° Elbow | 4 | EA | | 368 | | 530 | 898 |
| 14 | 4" Tee | 1 | EA | | 87 | | 130 | 217 |
| 15 | 4" Cross | 1 | EA | | 487 | | 174 | 661 |
| 16 | 4" x 2" Tee | 1 | EA | | 215 | | 130 | 345 |
| 17 | 6" x 4" Reducer | 2 | EA | | 68 | | 219 | 287 |
| 18 | 4" Flange | 17 | EA | | 2 533 | | 739 | 3 272 |
| 19 | 6" Flange | 3 | EA | | 834 | | 199 | 1 033 |
| 20 | Bolts and Gaskets | 1 | Lot | | 245 | | 511 | 756 |

MAT'L. $ 26,144 LABOR $ 7,060 TOTALS THIS SHEET

# SVERDRUP & PARCEL
# CONSTRUCTION COST ESTIMATE

PAGE ______

SHEET 3 SHEETS THIS SET 4

JOB NO. 7518 CLIENT EPA-IERL

PROJECT Dyebath Reuse

PHASE OR DIVISION Mechanical Piping/Instrumentation

BASIS FOR ESTIMATE
☐ NO DESIGN COMPLETED
☒ PRELIMINARY DESIGN
☐ FINAL DESIGN
☐

OFFICE St. Louis SECTION Cost Control BY MFF CHKD JDU DATE 10/10/83

| DESCRIPTION | QUANTITY |  | MATERIAL OR EQUIPMENT |  | LABOR OR LUMP SUM OR SUBCONTRACT |  | TOTAL COST |
|---|---|---|---|---|---|---|---|
|  | NO. UNITS | UNIT MEAS. | PER UNIT | COST | PER UNIT | COST |  |
| ½" Ball valve | 1 | EA |  | 46 |  | 21 | 67 |
| 2" Ball Valve | 1 | EA |  | 152 |  | 41 | 193 |
| 4" Butterfly valve | 1 | EA |  | 350 |  | 54 | 404 |
| 4" Check valve | 1 | EA |  | 945 |  | 85 | 1 030 |
| 4" On-off valve | 4 | EA |  | 4 740 |  | 455 | 5 195 |
| 6" Floor Penetration | 1 | EA |  | 75 |  | 166 | 241 |
| 6" Pipe Hanger | 10 | EA |  | 350 |  | 207 | 557 |
|  |  |  |  |  |  |  |  |
| SUBTOTAL - MECHANICAL |  |  |  |  |  |  |  |
| EQUIPMENT AND PIPING |  |  |  | 32 802 |  | 8 089 | 40 891 |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |
| Level switch in tank | 1 | EA |  | 2 035 |  | 172 | 2 207 |
| Instrument air connections | 4 | EA |  | 740 |  | 1 035 | 1 775 |
|  |  |  |  |  |  |  |  |
| SUBTOTAL INSTRUMENTATION |  |  |  | 2 775 |  | 1 207 | 3 982 |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |

# SVERDRUP & PARCEL
# CONSTRUCTION COST ESTIMATE

PAGE ______
SHEET 4 SHEETS THIS SET 4

JOB NO. 7518 CLIENT EPA-IERL

PROJECT Dyebath Reuse

PHASE OR DIVISION Electrical (Subcontract)

BASIS FOR ESTIMATE
☐ NO DESIGN COMPLETED
☒ PRELIMINARY DESIGN
☐ FINAL DESIGN
☐

OFFICE St. Louis SECTION Cost Control BY GH CHKD JDU DATE 10/10/83

| DESCRIPTION | QUANTITY |  | MATERIAL OR EQUIPMENT |  | LABOR OR LUMP SUM OR SUBCONTRACT |  | TOTAL COST |
|---|---|---|---|---|---|---|---|
|  | NO. UNITS | UNIT MEAS. | PER UNIT | COST | PER UNIT | COST |  |
| Connect 15 hp motor | 1 | EA |  | 10 |  | 49 | 59 |
| Motor Starter | 1 | EA |  | 673 |  | 58 | 731 |
| Local Control Panel | 1 | EA |  | 200 |  | 117 | 317 |
| Wiring, conduit, etc. | 1 | Lot |  | 400 |  | 562 | 962 |
| Cable to level switch | 100 | LF |  | 79 |  | 158 | 237 |
| Cable to beck controller | 100 | LF |  | 112 |  | 226 | 338 |
| Subtotal |  |  |  | 1 474 |  | 1 170 | 2 644 |
| Sales Tax (4%) |  |  |  | 59 |  | --- | 59 |
| Subtotal |  |  |  | 1 533 |  | 1 170 | 2 703 |
| 26½% OH&P |  |  |  | 406 |  | 310 | 716 |
| Subtotal |  |  |  | 1 939 |  | 1 480 | 3 419 |
| Mechanical Contractor Markup | 10% |  |  | 194 |  | 148 | 342 |
|  |  |  |  |  |  |  |  |
| TOTAL-ELECTRICAL SUBCONTRACT |  |  |  | 2 133 |  | 1 628 | 3 761 |

## TABLE X-4

## CAPITAL COST OF ANALYTICAL EQUIPMENT

| | |
|---|---|
| Spectrophotometer with Multiple Sample Chamber and Analog Output | $2,600 |
| Analog-to-Digital RS-232C Interface | 900 |
| Desk-top Computer with Printer, Keyboard, Display, one Floppy Disc Drive, Operating System, and BASIC Interpreter | 3,500 |
| Glassware (Cuvettes, Seperatory Funnels, Graduated Cylinders, plus Miscellaneous Supplies) | 500 |
| Total | $7,500 |

### Total Capital Cost

The total capital cost for the dyebath reconstitution system is summarized below:

| | |
|---|---|
| Pumping System | $63,000 |
| Analytical Equipment | 7,500 |
| Development Costs | 10,000 |
| Total Capital Costs | $80,500 |

## OPERATING COSTS

Estimates of the yearly operating and maintenance costs for the dyebath reconstitution system are summarized below:

| | |
|---|---|
| Pump and valve maintenance | $ 2,000 |
| Octanol, salt, cotton | 1,500 |
| Glassware | 200 |
| Electrical Power | 400 |
| Spectrophotometer/Computer Service | 900 |
| TOTAL | $ 5,000 |

Note that if a less expensive solvent than octanol (e.g., hexanol) were shown to be usable for dyestuff extraction, then significant cost savings could be realized. Octanol costs are $30/liter, as compared to hexanol at $6/liter.

## ECONOMIC ANALYSIS

Two analyses of the economic feasiblity of dyebath reconstitution are presented below. One is a simple payback period calculation, the other a return-on-investment analysis.

### Payback Period

A simple payback period analysis for dyebath reconstitution at Mill C-2 was conducted, based on two dyebecks dedicated to dyeing using dyebath reconstitution:

| | |
|---|---|
| Yearly Savings | $60,000 |
| Yearly O&M Cost | 5,000 |
| Net Savings | $55,000 |
| | |
| Total Capital Cost | $80,500 |

Payback period = 1.5 years (Total Capital/Net Savings)

The yearly savings are calculated by assuming a savings of $25 per dye cycle. A total of 1,200 dyeings per beck per year is used for the cost basis. Note that if the piping/pumping construction work were done in-house, at a savings of $12,000, the resulting payback period is decreased to 15 months (1.25 years).

### Return-On-Investment

The after tax return on investment was calculated in the following manner:

- Initial capital investment is $80,500
- Yearly savings are $60,000
- Yearly O&M costs are $5,000
- Equipment is depreciated by straight-line method over 10 years with no salvage value
- Income tax rate is 50 percent

A cash flow chart and return on investment calculations are presented in Table X-5. The after tax rate of return is 36.8 percent which is very attractive.

## MAXIMIZING SAVINGS BY DYE PROCEDURE CHANGES

Several factors were noted earlier as decreasing the savings realized through dyebath reuse: high dye liquor ratio, low auxiliary chemical value, and cool-down rinsing. From a practical standpoint, only one of these factors, the amount of cool-down water used, can be changed at this mill to enhance the savings from dyebath reuse.

## TABLE X-5

## RETURN ON INVESTMENT

| Year | Before Tax Cash Flow | Depreciation | Taxable Income | Income Tax | After Tax Cash Flow |
|---|---|---|---|---|---|
| 0 | -$80,500 | | | | |
| 1 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 2 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 3 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 4 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 5 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 6 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 7 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 8 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 9 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| 10 | + 55,000 | - 7,000 | + 48,000 | + 24,000 | + 31,000 |
| Totals | $469,500 | -70,000 | 480,000 | 240,000 | 310,000 |

Rate of return calculation:

$$\$\ 80{,}500 = \frac{(1 + i)^n - 1}{i\ (1+i)^n} * \$31{,}000$$

After tax rate of return (i) = 36.8 percent.

As discussed in Section VIII, the purpose of the rinse is to cool the carpet to 150°F or lower before dropping the dyebath. The rinse water, however, also dilutes the exhausted dyebath, thereby requiring more auxiliary chemicals to be added to reconstitute the bath. Therefore, the savings achievable by dyebath reuse can be maximized by minimizing the amount of cool-down water used.

Two alternatives were presented in Section VIII that would decrease the amount of cooling water required to about 1,500 gallons. These alternatives were to use a bump-and-run dyeing procedure, or simply to leave the steam shut off after carpet sampling, thus allowing the dyebeck to cool to about 165°F before the cool-down rinse.

The added cost savings per cycle that would result from either of these changes are estimated as follows:

| | Style 1 | Style 2 |
|---|---|---|
| Original Savings | $23.85 | $28.60 |
| Added Savings | | |
| Water/Sewer | 2.10 | 1.10 |
| Energy | -- | -- |
| Dye/Chemicals | 4.70 | 3.00 |
| TOTAL | $30.65 | $32.70 |

Energy savings are estimated to be very small.

Using a total of $30 per cycle savings versus the original $25 increases the annual savings by $12,000 to $72,000. This decreases the payback period to 1.2 years and raises the rate of return to 44.8 percent.

COMMENTS

Normally, a payback period of about 1 year or less is expected with dyebath reuse. At this mill, the higher liquor ratio used in dyeing carpets requires a large pumping/storage system, thereby increasing the capital cost. While the cost savings for water use and energy are also correspondingly greater due to the high liquor ratio, the auxiliary chemical savings, the largest area of cost savings, are not increased due to the higher liquor ratio.

Nevertheless, it is encouraging to note that at a mill with non-optimum conditions, and without optimizing the dye cycle to accommodate dyebath reuse, the resulting payback period and return on investment are sufficient to warrant the use of this wastewater recycle technology.

## REFERENCES

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12. Ewing, G. W. Instrumental Methods of Chemical Analysis, McGraw-Hill Book Company, 1960.

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# APPENDIX A

# DYEBATH REUSE PROGRAM LISTING

```
10  REM   DYEBATH REUSE PROGRAM - VERSION 3.0
20  REM   SOURCE LANGUAGE: MS-BASIC (Z-BASIC)
30  REM   WRITTEN:  1983
40  REM   BY:  SVERDRUP & PARCEL AND ASSOCIATES, INC.
50  REM        ST. LOUIS, MISSOURI
60  REM
70  REM   FOR: U.S. ENVIRONMENTAL PROTECTION AGENCY
80  REM        I.E.R.L.  (68-02-3678)
90  REM        RESEARCH TRIANGLE PARK, NC
100 REM
105 ON ERROR GOTO 12030
110 DEFINT F,I,J,L,M,N
120 OPTION BASE 1
130 DIM A(4),C(4),D(4),IROW(4),K(4,4),DYE$(4),GAMMA(4)
140 DIM STYLE$(10),DOWF(4),AUX$(7),AOWF(7),YNDYE(4),YNAUX(7)
150 DIM ODYE$(4),OAUX$(7),OAOWF(7),DLBS(10),AFACTOR(7)
160 DIM EXHAUST(4), ABSOR(4), AUXADD(7), ACON(7), OACON(7), UNIT$(7), OUNIT$(7)
170 FOR I=1 TO 10
180 READ STYLE$(I)
190 NEXT I
200 DATA "STYLE A1","STYLE B2","STYLE C3","STYLE D4","STYLE E5","STYLE F6"
210 DATA "STYLE G7","STYLE H8","STYLE I9","STYLE 10"
220 OPEN "R",#1,"A:RECIPES.DAT",24
230 FIELD #1,2 AS A$,4 AS B$,2 AS C$,4 AS E$,4 AS G$,4 AS I$,4 AS K$
240 OPEN "R",#2,"A:SYSTEMS.DAT",210
250 FIELD #2,2 AS CC$,2 AS NN$,2 AS X3$,5 AS A3$,5 AS B3$,5 AS C3$,5 AS D3$,184 AS X4$
260 FIELD #2,26 AS X5$,8 AS E3$,8 AS F3$,8 AS G3$,8 AS H3$,8 AS I3$,8 AS J3$,8 AS K3$,128 AS X6$
270 FIELD #2,82 AS X7$,2 AS A1$,2 AS B1$,2 AS C1$,2 AS D1$,120 AS X8$
280 FIELD #2,90 AS X9$,4ASA2$,4ASB2$,4ASC2$,4ASD2$,4ASE2$,4ASF2$,4ASG2$,4ASH2$,4ASI2$,4ASJ2$,4ASK2$
    ,4ASL2$,4ASM2$,4ASN2$,4ASO2$,4ASP2$,56 AS X10$
290 FIELD #2,154 AS X11$,4 AS O$,4 AS R$,4 AS T$,4 AS V$,4 AS X$,4 AS Z$,4 AS BB$,4 AS N$,4 AS P$,4
    AS S$,4 AS U$,4 AS W$,4 AS Y$,4 AS AA$
300 GOSUB 4900
310 DLBS(10)=0!
320 CLS
330 PRINT TAB(10) "DYEBATH RECONSTITUTION SYSTEM"
340 PRINT
350 PRINT TAB(20) "VERSION 3.0"
360 PRINT
370 PRINT
380 PRINT
390 PRINT TAB(5) "A.  REUSE DYEING"
400 PRINT
```

```
410 PRINT TAB(5) "B.  UPDATE RECIPES-DATA FILES"
420 PRINT
430 PRINT TAB(5) "C.  EXIT"
440 PRINT
450 PRINT
460 INPUT "ENTER CHOICE <A, B, or C> ";CHOICE$
470 IF CHOICE$ = "A" OR CHOICE$ = "a" THEN 510
480 IF CHOICE$ = "B" OR CHOICE$ = "b" THEN GOSUB 5400: GOTO 320
490 IF CHOICE$ = "C" OR CHOICE$ = "c" THEN 12010
500 GOTO 450
510 REM RECONSTITUTION MONITOR PROGRAM
520 CLS
530 OLDFLAG = 1!
540 PRINT "INPUT LAST STYLE AND SHADE DYED"
550 PRINT
560 FOR I=1 TO 10
570 PRINT I;"   ";STYLE$(I)
580 NEXT I
590 PRINT
600 INPUT "TYPE NUMBER OF STYLE  ",STYLENO%
610 IF STYLENO%<1 OR STYLENO%>10, THEN PRINT "INVALID ENTRY": GOTO 600
620 PRINT
630 PRINT
640 INPUT "INPUT SHADE NUMBER  ",SHADE
650 IF SHADE<1, THEN PRINT "INVALID ENTRY": GOTO 630
660 PRINT
670 PRINT
680 PRINT STYLE$(STYLENO%);"  SHADE";SHADE
690 INPUT "Correct <Y or N>";Q$
700 IF Q$ = "Y" OR Q$ = "y" GOTO 720
710 GOTO 520
720 PRINT: PRINT
730 INPUT "INPUT WEIGHT OF LAST DYEING (Pounds)  ",OWEIGHT
740 IF OWEIGHT<0, THEN PRINT "INVALID ENTRY": GOTO 720
750 PRINT OWEIGHT;"Pounds, ";
760 INPUT "Correct <Y or N>";Q$
770 IF Q$ = "Y" OR Q$ = "y" GOTO 790
780 GOTO 720
790 GOSUB 5100
800 OLDFLAG = 0!
```

```
810 CLS
820 INPUT "INPUT VOLUME OF DYEBATH TO BE REUSED (gallons)  ",VOLUME
830 IF VOLUME<0, THEN PRINT "INVALID ENTRY": GOTO 810
840 PRINT
850 PRINT VOLUME;"Gallons, ";
860 INPUT "Correct <Y or N>";Q$
870 IF Q$ = "Y" OR Q$ = "y" GOTO 890
880 GOTO 810
890 PRINT: PRINT: PRINT
900 INPUT "INPUT TOTAL VOLUME OF EXHAUSTED DYEBATH INCLUDING ANY OVERFLOW LOSSES (Gallons)  ",VOLUME1
910 IF VOLUME1 < VOLUME, THEN PRINT "INVALID ENTRY": GOTO 890
920 PRINT
930 PRINT VOLUME1; "Gallons, Correct <Y or N>";
940 INPUT Q$
950 IF Q$ = "Y" OR Q$ = "y" GOTO 970
960 GOTO 890
970 DFACTOR=1
980 REM  SPECTROPHOTOMETER INPUT ROUTINE
990 FOR I=1 TO N
1000 CLS
1010 PRINT
1020 PRINT "SET WAVELENGTH TO";GAMMA(I);"nm"
1030 PRINT
1040 PRINT "INSERT 'BLANK' CUVETTE AND SET ABSORBANCE AT 0.000"
1050 GOSUB 4300
1060 ABSOR(I)=A(I)
1070 NEXT I
1080 CLS
1090 REM  SOLVE FOR CONCENTRATIONS
1100 GOSUB 3500
1110 PRINT: PRINT "DYE","  CONC %","  EXHAUST %"
1120 PRINT "~~~~","  ~~~~~~~","  ~~~~~~~~~~"
1130 FOR I=1 TO N
1140 C(I)=C(I)*DFACTOR
1150 IF C(I) < 0, THEN C(I)=0
1160 EXHAUST(I) = 100!*(1-(C(I)*VOLUME1*8.34)/(DOWF(I)*OWEIGHT))
1170 PRINT DYE$(I),C(I),
1180 PRINT USING "#####.#"; EXHAUST(I)
1190 DLBS(I)=C(I)*VOLUME*8.340001E-02
1200 NEXT I
```

```
1210 PRINT
1220 GOSUB 4100
1230 INPUT "HIT 'RETURN' TO CONTINUE",ANY$
1240 CLS
1250 PRINT "INPUT NEXT STYLE AND SHADE"
1260 PRINT
1270 FOR I=1 TO 10
1280 PRINT I;"   ";STYLE$(I)
1290 NEXT I
1300 PRINT
1310 INPUT "TYPE NUMBER OF STYLE  ",STYLENO%
1320 IF STYLENO%<1 OR STYLENO%>10, THEN PRINT "INVALID ENTRY": GOTO 1310
1330 PRINT
1340 PRINT
1350 INPUT "INPUT SHADE NUMBER  ",SHADE
1360 IF SHADE<1, THEN PRINT "INVALID ENTRY": GOTO 1340
1370 PRINT
1380 PRINT
1390 PRINT STYLE$(STYLENO%);"  SHADE";SHADE
1400 INPUT "Correct <Y or N>";Q$
1410 IF Q$ = "Y" OR Q$ = "y" GOTO 1430
1420 GOTO 1240
1430 PRINT: PRINT
1440 INPUT "INPUT WEIGHT OF NEXT DYEING (Pounds)  ",WEIGHT
1450 IF WEIGHT<0, THEN PRINT "ILLEGAL ENTRY": GOTO 1430
1460 PRINT WEIGHT;"Pounds, ";
1470 INPUT "Correct <Y or N>";Q$
1480 IF Q$ = "Y" OR Q$ = "y" GOTO 1500
1490 GOTO 1430
1500 GOSUB 5100
1560 GOSUB 1600
1570 GOSUB 2000
1580 GOTO 320
1590 END
```

```
1600 REM  AUXILIARY CHEMICAL INVENTORY
1610 CLS
1620 PRINT "WERE ANY ADDITIONAL AUXILIARY CHEMICALS ADDED DURING"
1630 INPUT "THE PREVIOUS DYE CYCLE (Chemical Adds) <Y or N>";Q$
1640 IF Q$="Y" OR Q$="y" GOTO 1670
1650 IF Q$="N" OR Q$="n", THEN RETURN
1660 GOTO 1610
1670 CLS
1680 PRINT "INPUT QUANTITIES ADDED"
1690 PRINT
1700 FOR I=1 TO 7
1710 IF DAUX$(I)="       " THEN 1740
1720 PRINT DAUX$(I);" ";OUNIT$(I);
1730 INPUT " ",AUXADD(I)
1740 NEXT I
1750 CLS
1760 PRINT "AUXILIARIES ADDED"
1770 PRINT
1780 FOR I=1 TO 7
1790 IF DAUX$(I)="       " THEN 1810
1800 PRINT DAUX$(I);" ";AUXADD(I);" ";OUNIT$(I)
1810 NEXT I
1820 PRINT
1830 INPUT "Correct <Y or N>";Q$
1840 IF Q$="Y" OR Q$="y" THEN 1860
1850 GOTO 1670
1860 FOR I=1 TO 7
1870 IF OUNIT$(I)="gal", THEN AUXADD(I)=AUXADD(I)*DACON(I)
1880 NEXT I
1890 RETURN
1900 END
```

```
2000 REM  COMPUTE AND PRINT ADD QUANTITIES
2010 GOSUB 3100
2020 PRINT "PRINTING..."
2030 JOBDT$=DATE$
2040 JOBTM$=TIME$
2050 LPRINT TAB(30) CHR$(14) CHR$(27) CHR$(69) "EPA - IERL"
2060 LPRINT TAB(25) CHR$(14) "CARPET MILL C-2"
2070 LPRINT TAB(30) CHR$(27) CHR$(70) CHR$(15) CHR$(27) CHR$(71);
2080 LPRINT CHR$(14) "SVERDRUP & PARCEL"
2090 LPRINT CHR$(18) CHR$(27) CHR$(72)
2100 LPRINT CHR$(10) TAB(70) JOBDT$
2110 LPRINT TAB(71) JOBTM$
2120 LPRINT CHR$(10)
2130 LPRINT CHR$(10) "PRESENT ";
2140 LPRINT "STYLE: " CHR$(27) CHR$(45) CHR$(1) STYLE$(STYLENO%);
2150 LPRINT CHR$(27) CHR$(45) CHR$(0) TAB(35);
2160 LPRINT "SHADE:" CHR$(27) CHR$(45) CHR$(1) SHADE;
2170 LPRINT CHR$(27) CHR$(45) CHR$(0) TAB(53);
2180 LPRINT "WEIGHT: " CHR$(27) CHR$(45) CHR$(1) WEIGHT;
2190 LPRINT CHR$(27) CHR$(45) CHR$(0) "lbs"
2200 LPRINT CHR$(10) "PREVIOUS: " TAB(16) STYLE$(OSTYLE%) TAB(39) OSHD TAB(57) OWEIGHT "lbs"
2210 LPRINT CHR$(10) "VOLUME RECYCLED:" VOLUME "GALLONS"
2220 LPRINT CHR$(10) CHR$(10) "ADD QUANTITIES"
2230 LPRINT CHR$(10) CHR$(27) CHR$(45) CHR$(1) "DYESTUFF" TAB(15) "RECYCLE";
2240 LPRINT TAB(26) "RECIPE" TAB(37) "ADD" CHR$(27) CHR$(45) CHR$(0) CHR$(10)
2250 FOR I=1 TO 4
2260 YNDYE(I)=0!
2270 NEXT I
2280 FOR I=1 TO N
2290 NAM$=DYE$(I)
2300 RECLB=DOWF(I)*WEIGHT*.01
2310 FOR J=1 TO N
2320 IF ODYE$(J)=NAM$, THEN FLAG=J: YNDYE(J)=1!: GOTO 2350
2330 NEXT J
2340 FLAG=10
2350 ADDDYE=RECLB-DLBS(FLAG)
2360 IPOUND=FIX(ADDDYE)
2370 ADDOZ=(ADDDYE-IPOUND)*16!
2380 IOUNCE=FIX(ADDOZ)
2390 IGRAIN=FIX((ADDOZ-IOUNCE)*437.5)
2400 LPRINT NAM$ TAB(11);
2410 LPRINT USING "#######.##"; DLBS(FLAG),RECLB;
2420 LPRINT SPC(3) IPOUND "lbs," IOUNCE "oz," IGRAIN "gr";
2430 IF ADDDYE < 0, THEN LPRINT TAB(70) "****";
2440 LPRINT " "
2450 NEXT I
```

```
2460 FOR J=1 TO OLDN%
2470 IF YNDYE(J) > .5 GOTO 2510
2480 LPRINT ODYE$(J) TAB(11);
2490 LPRINT USING "#######.##";DLBS(J);
2500 LPRINT TAB(70) "****"
2510 NEXT J
2520 LPRINT CHR$(10) CHR$(10) CHR$(27) CHR$(45) CHR$(1) "AUXILIARY";
2530 LPRINT TAB(15) "RECYCLE" TAB(26) "RECIPE" TAB(37) "ADD";
2540 LPRINT CHR$(27) CHR$(45) CHR$(0) CHR$(10)
2550 FOR I=1 TO 7
2560 YNAUX(I)=0!
2570 NEXT I
2580 FOR I=1 TO 7
2590 NAM$=AUX$(I)
2600 IF NAM$="        " GOTO 2750
2610 RECLB=AOWF(I)*WEIGHT*.01
2620 FOR J=1 TO 7
2630 IF OAUX$(J)=NAM$, THEN FLAG=J: YNAUX(J)=1!: GOTO 2670
2640 NEXT J
2650 RCYLB=0!
2660 GOTO 2680
2670 RCYLB=(OAOWF(FLAG)*OWEIGHT*.01+AUXADD(FLAG))*AFACTOR(FLAG)
2680 ADDAUX=RECLB-RCYLB
2690 IF UNIT$(I)="gal", THEN ADDAUX=ADDAUX/ACON(I): RECLB=RECLB/ACON(I):RCYLB=RCYLB/ACON(I)
2700 LPRINT NAM$ TAB(11);
2710 LPRINT USING "#######.##";RCYLB,RECLB;ADDAUX;
2720 LPRINT " ";UNIT$(I);
2730 IF ADDAUX < 0, THEN LPRINT TAB(70) "****";
2740 LPRINT " "
2750 NEXT I
2760 FOR J=1 TO 7
2770 IF YNAUX(J) > .5 GOTO 2840
2780 IF OAUX$(J)="        " GOTO 2840
2790 RCYLB=(OAOWF(J)*OWEIGHT*.01 + AUXADD(J))*AFACTOR(J)
2800 IF OUNIT$(J)="gal" THEN RCYLB = RCYLB/OACON(J)
2810 LPRINT OAUX$(J) TAB(11);
2820 LPRINT USING "#######.##";RCYLB;
2830 LPRINT TAB(70) "****"
2840 NEXT J
2850 GOSUB 2900
2860 LPRINT CHR$(140)
2870 RETURN
2880 END
```

```
2900 REM PRINT EXPERIMENTAL DATA
2910 LPRINT CHR$(10) CHR$(10) TAB(32) CHR$(27) CHR$(45) CHR$(1);
2920 LPRINT "EXPERIMENTAL DATA" CHR$(27) CHR$(45) CHR$(0)
2930 LPRINT CHR$(10) "WAVELENGTH" TAB(15) "ABSORBANCE" TAB(40) "DYESTUFF";
2940 LPRINT TAB(53) "CONCENTRATION %" TAB(72) "EXHAUST %"
2950 LPRINT
2960 FOR I=1 TO N
2970 LPRINT TAB(3) GAMMA(I) TAB(18);
2980 LPRINT USING "##.###";ABSOR(I);
2990 LPRINT TAB(41) ODYE$(I) TAB(53) C(I) TAB(73);
3000 LPRINT USING "###.##";EXHAUST(I)
3010 NEXT I
3020 RETURN
3030 END
```

```
3100 REM  AUXILIARY LOSS CALCULATIONS
3110 CLS
3112 ALOSS = VOLUME/VOLUME1
3115 LOSS = CINT(100*(1!-ALOSS))
3120 PRINT "THE SYSTEM WILL ASSUME A"; LOSS; "% LOSS FOR ALL AUXILIARIES."
3130 PRINT "DO YOU WISH TO CHANGE THIS TO A DIFFERENT LOSS PERCENTAGE <Y or N>";
3140 INPUT Q$
3150 IF Q$ = "N" OR Q$ = "n", THEN ALOSS = 1!-LOSS/100!: GOTO 3240
3160 PRINT
3170 INPUT "TYPE NEW LOSS PERCENTAGE <0 to 100>  ", ANLOSS
3180 IF ANLOSS > 100 OR ANLOSS < 0, THEN PRINT "INVALID ENTRY": GOTO 3160
3190 PRINT
3200 PRINT ANLOSS "PERCENT <Y or N>";
3210 INPUT Q$
3220 IF Q$ = "Y" OR Q$ = "y", THEN ALOSS = 1!-ANLOSS/100!: GOTO 3240
3230 GOTO 3110
3240 FOR I=1 TO 7
3250 AFACTOR(I) = ALOSS
3260 NEXT I
3270 CLS
3280 PRINT "THE SYSTEM WILL ASSUME THE FOLLOWING LOSSES FOR THE"
3290 PRINT "AUXILIARY CHEMICALS IN THE LAST DYEBATH:"
3300 PRINT
3310 FOR I=1 TO 7
3320 IF DAUX$(I)="        " THEN 3350
3330 PRINT I DAUX$(I);"  ";
3340 PRINT USING "###.#"; (1 - AFACTOR(I))*100;: PRINT "%"
3350 NEXT I
3360 PRINT
3370 PRINT "TO MAKE ANY CHANGES, TYPE NUMBER OF AUXILIARY CHEMICAL"
3380 PRINT "HIT RETURN TO CONTINUE"
3390 INPUT S6%
3400 IF S6% > 7 OR S6% < 0, THEN PRINT "INVALID ENTRY": GOTO 3370
3410 IF S6% = 0 GOTO 3460
3420 INPUT "TYPE NEW LOSS PERCENTAGE <0 to 100>  ", S7
3430 IF S7<0 OR S7>100, THEN PRINT "INVALID ENTRY": GOTO 3420
3440 AFACTOR(S6%)=1! - (S7/100!)
3450 GOTO 3270
3460 RETURN
3470 END
```

```
3500 REM  SOLVE N x N MATRIX
3510 FOR I= 1 TO N
3520 IROW(I)=I
3530 D(I)=0!
3540 FOR J=1 TO N
3550 IF D(I)>=ABS(K(I,J)) THEN 3570
3560 D(I)=ABS(K(I,J))
3570 NEXT J
3580 NEXT I
3590 NM=N-1
3600 FOR IDIAG=1 TO NM
3610 L=IDIAG+1
3620 ID=IROW(IDIAG)
3630 BIG=ABS(K(ID,IDIAG))/D(ID)
3640 IBIG=IDIAG
3650 FOR I=L TO N
3660 IR=IROW(I)
3670 T=ABS(K(IR,IDIAG))/D(IR)
3680 IF BIG>=T THEN 3710
3690 BIG=T
3700 IBIG=I
3710 NEXT I
3720 IF IBIG=IDIAG THEN 3750
3730 IROW(IDIAG)=IROW(IBIG)
3740 IROW(IBIG)=ID
3750 ID=IROW(IDIAG)
3760 FOR I=L TO N
3770 IR=IROW(I)
3780 T=K(IR,IDIAG)/K(ID,IDIAG)
3790 K(IR,IDIAG)=T
3800 FOR J=L TO N
3810 K(IR,J)=K(IR,J)-K(ID,J)*T
3820 NEXT J
3830 A(IR)=A(IR)-A(ID)*K(IR,IDIAG)
3840 NEXT I
3850 NEXT IDIAG
3860 K%=IROW(N)
3870 C(N)=A(K%)/K(K%,N)
3880 M=N-1
3890 L=N
3900 FOR J=1 TO M
3910 SUM=0!
3920 K%=IROW(L-1)
3930 FOR I=L TO N
3940 SUM=SUM+(K(K%,I))*C(I)
3950 NEXT I
3960 L=L-1
3970 C(L)=(A(K%)-SUM)/K(K%,L)
3980 NEXT J
3990 RETURN
4000 END
```

```
4100 REM  SAVE PREVIOUS STYLE/SHADE DATA
4110 OSTYLE%=STY%
4120 OSHD=SHD
4130 FOR I=1 TO N
4140 ODYE$(I) = DYE$(I)
4150 NEXT I
4160 FOR I=1 TO 7
4170 OAUX$(I) = AUX$(I)
4180 OAOWF(I) = AOWF(I)
4190 OUNIT$(I)=UNIT$(I)
4200 OACON(I)=ACON(I)
4210 NEXT I
4220 OLDN% = N
4230 OLDSYS%=SYSNO%
4240 RETURN
4250 END
```

```
4300 REM   DATA INPUT FROM SPEC-21
4310 BEEP
4320 INPUT "HIT 'RETURN' WHEN READY TO CONTINUE",ANY$
4330 IF LOC(3)=0 THEN 4350
4340 VOID$=INPUT$(LOC(3),#3)
4350 PRINT #3,"BCD.5"
4360 WHILE LOC(3)<62
4370 WEND
4380 PRINT #3,CHR$(0)
4390 INPUT #3,VOID$,VOID1$,VOID2$,VOID3$,VOID4$,VOID5$,VOID6$,IZERO
4400 IF IZERO=0 THEN 4780
4410 PRINT
4420 PRINT
4430 BEEP
4440 PRINT "INSERT SAMPLE CUVETTE"
4450 INPUT "HIT 'RETURN' WHEN READY",ANY$
4460 IF LOC(3)=0 THEN 4480
4470 VOID$=INPUT$(LOC(3),#3)
4480 PRINT #3,"BCD.5"
4490 WHILE LOC(3)<62
4500 WEND
4510 PRINT #3,CHR$(0)
4520 INPUT #3,VOID$,VOID1$,VOID2$,VOID3$,VOID4$,VOID5$,VOID6$,ITRN
4530 TRANS=ITRN*100!/IZERO
4540 IF TRANS=0, THEN 4780
4550 A(I)=2!-(LOG(TRANS))/2.3026
4560 IF A(I)<0!,THEN A(I)=0!
4570 PRINT
4580 PRINT
4590 PRINT "ABSORBANCE AT"; GAMMA(I); " nm = ";
4600 PRINT USING "#.###"; A(I)
4610 INPUT "Correct <Y or N>";Q$
4620 IF Q$="Y" OR Q$="y" THEN 4640
4630 INPUT "INPUT ABSORBANCE  ",A(I)
4640 IF A(I)>1.2 THEN 4700
4650 RETURN
4660 END
```

*** SEE NEXT PAGE FOR NO-INTERFACE ALTERNATE ***

```
*** NO-INTERFACE ALTERNATE ***


4300 REM   DATA INPUT FROM SPEC-21
4310 BEEP
4320 INPUT "HIT 'RETURN' WHEN READY TO CONTINUE",ANY$
4410 PRINT
4420 PRINT
4430 BEEP
4440 PRINT "INSERT SAMPLE CUVETTE"
4450 INPUT "TYPE IN ABSORBANCE AND HIT 'RETURN'  ",A(I)
4570 PRINT
4580 PRINT
4590 PRINT "ABSORBANCE AT"; GAMMA(I); " nm = ";
4600 PRINT USING "#.###"; A(I)
4610 INPUT "Correct <Y or N>";Q$
4620 IF Q$="Y" OR Q$="y" THEN 4640
4630 GOTO 4450
4640 IF A(I)>1.2 THEN 4700
4650 RETURN
4660 END
```

```
4700 REM  DILUTION ROUTINE
4710 PRINT
4720 BEEP
4730 PRINT "ABSORBANCE IS TOO HIGH"
4740 PRINT "DILUTE 1 PART SAMPLE WITH 1 PART BLANK LIQUOR <or solvent>"
4750 DFACTOR = DFACTOR*2!
4760 INPUT "HIT 'RETURN' WHEN READY TO CONTINUE",ANY$
4770 GOTO 980
4780 PRINT "---DATA READ ERROR---"
4790 PRINT "CHECK SPECTROPHOTOMETER AND/OR INTERFACE"
4800 GOTO 12030
4810 END
```

```
4900 REM  INTERFACE SET-UP ROUTINE
4910 CLS
4920 BEEP
4930 PRINT "SET INTERFACE 'ON/OFF' POWER SWITCH TO 'ON'"
4940 INPUT "HIT 'RETURN' WHEN READY TO CONTINUE",ANY$
4950 OPEN "COM1:1200,E,7,2" AS #3
4960 PRINT #3,"RANGE1 4"
4970 PRINT #3,"RANGE2 4"
4980 IF LOC(3)<>0 THEN 5000
4990 RETURN
5000 SPEC$=INPUT$(LOC(3),#3)
5010 PRINT SPEC$;
5020 GOTO 4980
5030 END
```

*** NO-INTERFACE ALTERNATE ***

```
4900 REM  INTERFACE SET-UP ROUTINE
4910 CLS
4990 RETURN
5030 END
```

```
5100 REM  READ RECIPE AND SYSTEM FILES
5110 PRINT "SEARCHING FILES..."
5120 I=1
5130 GET #1,I
5140 IF EOF(1) THEN 5320
5150 STY%=CVI(A$)
5160 IF STY%=STYLENO% THEN 5190
5170 I=I+1
5180 GOTO 5130
5190 SHD=CVS(B$)
5200 IF ABS(SHD-SHADE)<.001 THEN 5230
5210 I=I+1
5220 GOTO 5130
5230 GOSUB 11200
5240 IF OLDFLAG < .5 AND OLDSYS%=SYSNO%, THEN RETURN
5250 GET #2,SYSNO%
5260 IF EOF(2) THEN 5350
5270 GOSUB 11300
5280 IF OLDFLAG < .5, THEN RETURN
5290 GOSUB 11700
5300 RETURN
5310 REM  ERROR RETURNS
5320 PRINT "CAN'T FIND STYLE/SHADE"
5330 INPUT "HIT 'RETURN' TO START OVER ",ANY$
5340 RETURN 320
5350 PRINT "CAN'T FIND SYSTEM"
5360 INPUT "HIT 'RETURN' TO START OVER ",ANY$
5370 RETURN 320
5380 END
```

```
5400 REM  EDIT RECIPE FILE
5410 CLS
5420 PRINT "WHAT DO YOU WANT TO DO?"
5430 PRINT
5440 PRINT "A.  ADD RECIPE"
5450 PRINT
5460 PRINT "B.  REVIEW OR CHANGE EXISTING RECIPE"
5470 PRINT
5480 PRINT "C.  LIST ALL RECIPES"
5490 PRINT
5500 PRINT "D.  REUSE DYEING OR EXIT"
5510 PRINT
5520 PRINT "ENTER CHOICE <A to D>";
5530 INPUT CHOICE$
5540 IF CHOICE$="A" OR CHOICE$="a", THEN GOSUB 5600: GOTO 5410
5550 IF CHOICE$="B" OR CHOICE$="b", THEN GOSUB 6000: GOTO 5410
5560 IF CHOICE$="C" OR CHOICE$="c", THEN GOSUB 10800: GOTO 5410
5570 IF CHOICE$="D" OR CHOICE$="d", THEN RETURN
5580 GOTO 5520
5590 END
```

```
5600 REM  ADD RECIPE
5610 GOSUB 6800
5620 IRECNO=0
5630 GOSUB 7100
5640 IF IRECNO>0, THEN PRINT "RECIPE ALREADY EXISTS": INPUT "HIT 'RETURN' TO CONTINUE",ANY$: RETURN
5650 IRECNO=ILASTREC
5660 GOSUB 8300
5670 FOR I=1 TO 4
5680 DOWF(I)=0!
5690 NEXT I
5700 GOSUB 8100
5710 GOSUB 6600
5720 CLS
5730 PRINT "DO YOU WISH TO ADD ANY MORE RECIPES OF THIS STYLE"
5740 INPUT "USING THE SAME DYE SYSTEM <Y or N>";Q$
5750 IF Q$="Y" OR Q$="y" THEN 5770
5760 RETURN
5770 INPUT "NEXT SHADE NUMBER  ",SHADE
5780 IF SHADE < 1, THEN PRINT "INVALID ENTRY": GOTO 5720
5790 PRINT
5800 PRINT
5810 PRINT STYLE$(STYLENO%);"  SHADE";SHADE
5820 PRINT "Correct <Y or N> ";
5830 INPUT Q$
5840 IF Q$="Y" OR Q$="y" THEN 5860
5850 GOTO 5720
5860 IRECNO=0
5870 GOSUB 7100
5880 IF IRECNO>0, THEN PRINT "RECIPE ALREADY EXISTS": INPUT "HIT 'RETURN' TO CONTINUE",ANY$: RETURN
5890 IRECNO=ILASTREC
5900 GOTO 5670
5910 END
```

```
6000 REM  REVIEW OR CHANGE RECIPE
6010 GOSUB 6800
6020 IRECNO=0
6030 GOSUB 7100
6040 IF IRECNO=0, THEN PRINT "RECIPE NOT FOUND": INPUT "HIT 'RETURN' TO CONTINUE",ANY$: RETURN
6050 GOSUB 11200
6060 GET #2,SYSNO%
6070 IF EOF(2) THEN 6530
6080 GOSUB 11300
6090 GOSUB 11700
6100 GOSUB 8100
6110 LOCATE 18,1,1
6120 INPUT "DO ANY DYE NAMES OR AUXILIARY NAMES CHANGE <Y or N> ";Q$
6130 IF Q$="Y" OR Q$="y", THEN GOSUB 9500: GOTO 6160
6140 INPUT "DO AUXILIARY CHEMICAL WEIGHTS CHANGE <Y or N> ";Q$
6150 IF Q$="Y" OR Q$="y", THEN GOSUB 9500
6160 GOSUB 8100
6170 LOCATE 22,1,1
6180 INPUT "DO DYE WEIGHTS CHANGE <Y or N> ";Q$
6190 IF Q$="Y" OR Q$="y", THEN 6210
6200 GOSUB 6710: GOTO 6230
6210 GOSUB 6600
6230 INPUT "SHOULD ALL OTHER SHADES IN THIS SYSTEM BE REVIEWED FOR CHANGES ";Q$
6240 IF Q$="Y" OR Q$="y" THEN 6260
6250 RETURN
6260 IC=1
6270 GET #1,IC
6280 IF EOF(1) THEN RETURN
6285 SHADE = CVS(B$)
6290 GOSUB 11200
6300 IF SYSNO%=SYS% THEN 6330
6310 IC=IC+1
6320 GOTO 6270
6330 IRECNO=IC
6340 GOSUB 8100
6350 GOSUB 6600
6500 IC=IC+1
6510 GOTO 6270
6520 RETURN
6530 PRINT "ERROR - DYE SYSTEM NOT FOUND"
6540 INPUT "HIT 'RETURN' TO CONTINUE", ANY$
6550 RETURN
6560 END
```

```
6600 REM  CHANGE DYESTUFF O.W.F.
6610 LOCATE 22,1,1
6620 PRINT "TYPE NEW DYESTUFF o.w.f. AND 'RETURN' FOR EACH LINE"
6630 PRINT "OR HIT 'RETURN' ONLY FOR NO CHANGE"
6640 FOR I=1 TO 4
6650 IF DYE$(I)="     " THEN 6700
6660 LOCATE (2+I),32,1
6670 INPUT CHANGES$
6680 IF CHANGES$="" THEN 6700
6690 DOWF(I)=VAL(CHANGES$)
6700 NEXT I
6710 GOSUB 8100
6720 LOCATE 23,1,1
6730 INPUT "CORRECT <Y or N> ";Q$
6740 IF Q$="Y" OR Q$="y" THEN 6760
6745 GOSUB 8100
6750 GOTO 6610
6760 GOSUB 7300
6770 RETURN
6775 END
```

```
6800 REM  GET  STYLE AND SHADE
6810 CLS
6820 PRINT "INPUT STYLE AND SHADE"
6830 PRINT
6840 FOR I=1 TO 10
6850 PRINT I;"   ";STYLE$(I)
6860 NEXT I
6870 PRINT
6880 INPUT "TYPE NUMBER OF STYLE  ",STYLENO%
6890 IF STYLENO%<1 OR STYLENO%>10, THEN PRINT "INVALID ENTRY": GOTO 6870
6900 PRINT
6910 PRINT
6920 INPUT "INPUT SHADE NUMBER  ",SHADE
6930 IF SHADE<1, THEN PRINT "INVALID ENTRY": GOTO 6910
6940 PRINT
6950 PRINT
6960 PRINT STYLE$(STYLENO%);"  SHADE";SHADE
6970 PRINT "Correct <Y or N>";
6980 INPUT Q$
6990 IF Q$ = "Y" OR Q$ = "y", THEN RETURN
7000 GOTO 6810
7010 END
```

```
7100 REM  READ FILE UNTIL CORRECT RECIPE FOUND
7110 I=1
7120 GET #1,I
7130 IF EOF(1) THEN 7240
7140 STY%=CVI(A$)
7145 IF STY%=0 THEN 7240
7150 IF STY%=STYLENO% THEN 7180
7160 I=I+1
7170 GOTO 7120
7180 SHD=CVS(B$)
7190 IF ABS(SHD-SHADE)<.001 THEN 7220
7200 I=I+1
7210 GOTO 7120
7220 IRECNO=I
7230 RETURN
7240 ILASTREC=I
7250 RETURN
7260 END
```

```
7300 REM  OUTPUT RECIPE RECORD
7310 LSET A$=MKI$(STYLENO%)
7320 LSET B$=MKS$(SHADE)
7330 LSET C$=MKI$(SYSNO%)
7340 LSET E$=MKS$(DOWF(1))
7350 LSET G$=MKS$(DOWF(2))
7360 LSET I$=MKS$(DOWF(3))
7370 LSET K$=MKS$(DOWF(4))
7380 PUT #1,IRECNO
7390 RETURN
7400 END
```

```
7500 REM  OUTPUT SYSTEM RECORD
7510 LSET CC$=MKI$(SYS%)
7520 LSET X3$=MKI$(STYLENO%)
7530 LSET NN$=MKI$(N)
7540 LSET A1$=MKI$(GAMMA(1))
7550 LSET B1$=MKI$(GAMMA(2))
7560 LSET C1$=MKI$(GAMMA(3))
7570 LSET D1$=MKI$(GAMMA(4))
7580 LSET A2$=MKS$(K(1,1))
7590 LSET B2$=MKS$(K(1,2))
7600 LSET C2$=MKS$(K(1,3))
7610 LSET D2$=MKS$(K(1,4))
7620 LSET E2$=MKS$(K(2,1))
7630 LSET F2$=MKS$(K(2,2))
7640 LSET G2$=MKS$(K(2,3))
7650 LSET H2$=MKS$(K(2,4))
7660 LSET I2$=MKS$(K(3,1))
7670 LSET J2$=MKS$(K(3,2))
7680 LSET K2$=MKS$(K(3,3))
7690 LSET L2$=MKS$(K(3,4))
7700 LSET M2$=MKS$(K(4,1))
7710 LSET N2$=MKS$(K(4,2))
7720 LSET O2$=MKS$(K(4,3))
7730 LSET P2$=MKS$(K(4,4))
7740 LSET O$=MKS$(AOWF(1))
7750 LSET R$=MKS$(AOWF(2))
7760 LSET T$=MKS$(AOWF(3))
7770 LSET V$=MKS$(AOWF(4))
7780 LSET X$=MKS$(AOWF(5))
7790 LSET Z$=MKS$(AOWF(6))
7800 LSET BB$=MKS$(AOWF(7))
7810 LSET N$=MKS$(ACON(1))
```

```
7820 LSET P$=MKS$(ACON(2))
7830 LSET S$=MKS$(ACON(3))
7840 LSET U$=MKS$(ACON(4))
7850 LSET W$=MKS$(ACON(5))
7860 LSET Y$=MKS$(ACON(6))
7870 LSET AA$=MKS$(ACON(7))
7880 LSET A3$=DYE$(1)
7890 LSET B3$=DYE$(2)
7900 LSET C3$=DYE$(3)
7910 LSET D3$=DYE$(4)
7920 LSET E3$=AUX$(1)
7930 LSET F3$=AUX$(2)
7940 LSET G3$=AUX$(3)
7950 LSET H3$=AUX$(4)
7960 LSET I3$=AUX$(5)
7970 LSET J3$=AUX$(6)
7980 LSET K3$=AUX$(7)
7990 PUT #2,SYS%
8000 RETURN
8010 END
```

```
8100 REM  PRINT RECIPE
8110 CLS
8120 PRINT "STYLE: "; STYLE$(STYLENO%);"    SHADE: ";SHADE
8130 PRINT
8140 FOR I=1 TO 4
8150 IF DYE$(I)="    ", THEN PRINT: GOTO 8190
8160 PRINT DYE$(I) TAB(16);
8170 PRINT USING "##.####"; DOWF(I);
8180 PRINT " % o.w.f."
8190 NEXT I
8200 FOR I=1 TO 7
8210 IF AUX$(I)="       ", THEN PRINT: GOTO 8250
8220 PRINT AUX$(I) TAB(16);
8230 PRINT USING "##.##"; AOWF(I);
8240 PRINT " % o.w.f."
8250 NEXT I
8260 RETURN
8270 END
```

```
8300 REM  REVIEW DYE SYSTEMS
8310 CLS
8320 PRINT "PLEASE REVIEW THE FOLLOWING DYE SYSTEMS AND SELECT THE CORRECT ONE"
8330 PRINT
8340 INPUT "HIT 'RETURN' TO CONTINUE",ANY$
8350 ID=1
8360 GET #2,ID
8370 IF EOF(2) THEN 8560
8380 SYSTY%=CVI(X3$)
8385 IF SYSTY%=0 THEN 8560
8390 IF SYSTY%=STYLENO% THEN 8420
8400 ID=ID+1
8410 GOTO 8360
8420 GOSUB 11300
8430 GOSUB 11700
8440 GOSUB 10600
8450 PRINT: PRINT
8460 INPUT "ARE THESE THE CORRECT DYESTUFFS AND K-VALUES <Y or N> ";Q$
8470 IF Q$="Y" OR Q$="y" THEN 8500
8480 ID=ID+1
8490 GOTO 8360
8500 GOSUB 10500
8510 PRINT: PRINT
8520 INPUT "ARE THESE THE CORRECT AUXILIARY CHEMICALS AND % o.w.f. <Y or N> ";Q$
8530 IF Q$="Y" OR Q$="y" THEN 8570
8540 ID=ID+1
8550 GOTO 8360
8560 LASTREC=ID: GOSUB 8600
8570 SYSNO%=SYS%
8580 RETURN
8590 END
```

```
8600 REM  ADD NEW DYE SYSTEM
8610 CLS
8620 PRINT "MUST ADD NEW DYE SYSTEM"
8630 PRINT
8640 INPUT "INPUT NUMBER OF DYES <1 to 4>";N
8650 IF N<1 OR N>4 THEN PRINT "INVALID ENTRY": PRINT: GOTO 8640
8660 FOR I=1 TO 4
8670 IF I>N THEN DYE$(I)="     ": GAMMA(I)=0: GOTO 8750
8680 PRINT: PRINT "DYESTUFF NAME #";I;
8690 INPUT DYE$(I)
8700 IF LEN(DYE$(I))>5 THEN DYE$(I)=LEFT$(DYE$(I),5): GOTO 8720
8710 IF LEN(DYE$(I))<5 THEN DYE$(I)=DYE$(I)+STRING$((5-LEN(DYE$(I)))," ")
8720 PRINT DYE$(I);
8730 INPUT " CORRECT <Y or N>";Q$
8740 IF Q$="Y" OR Q$="y" THEN 8750 ELSE GOTO 8680
8750 NEXT I
8760 PRINT: PRINT
8770 FOR I=1 TO N
8780 PRINT: PRINT "GAMMA #";I;
8790 INPUT GAMMA(I)
8800 PRINT GAMMA(I);"nm";
8810 INPUT " CORRECT <Y or N>";Q$
8820 IF Q$="Y" OR Q$="y" THEN 8830 ELSE GOTO 8780
8830 NEXT I
8840 FOR I=1 TO 4
8850 FOR J=1 TO 4
8860 K(I,J)=0!
8870 NEXT J
8880 NEXT I
8890 GOSUB 10600
8900 PRINT: PRINT: PRINT "INPUT K-VALUES"
8910 FOR I=1 TO N
8920 FOR J=1 TO N
8930 LOCATE (2+I),(8*J+15),1
8940 INPUT "",K(J,I)
8950 NEXT J
8960 LOCATE (2+I),1,1: PRINT STRING$(80," "): LOCATE (2+I),1,1
8970 PRINT TAB(2);DYE$(I);TAB(20);
8980 PRINT USING "#####.##";K(1,I);K(2,I);K(3,I);K(4,I);
8990 INPUT " CORRECT <Y or N>";Q$
9000 IF Q$="Y" OR Q$="y" THEN 9010 ELSE GOTO 8920
9010 NEXT I
9020 CLS
9030 INPUT "TYPE NUMBER OF AUXILIARY CHEMICALS <0 to 7> ";NUM
```

```
9040 IF NUM<0 OR NUM>7 THEN PRINT "INVALID ENTRY": PRINT: GOTO 9030
9050 FOR I=1 TO 7
9060 IF I > NUM, THEN AUX$(I)="        ": ACON(I)=0!: GOTO 9230
9070 PRINT: PRINT
9080 PRINT "AUXILIARY #";I;
9090 INPUT AUX$(I)
9100 IF LEN(AUX$(I))>8 THEN AUX$(I)=LEFT$(AUX$(I),8): GOTO 9120
9110 IF LEN(AUX$(I))<8 THEN AUX$(I)=AUX$(I)+STRING$(8-LEN(AUX$(I))," ")
9120 INPUT "UNITS <1 for POUNDS, 2 for GALLONS>";UNITS
9130 IF UNITS=1 THEN UNIT$(I)="lbs": ACON(I)=0!: GOTO 9170
9140 IF UNITS=2 THEN UNIT$(I)="gal": GOTO 9160
9150 GOTO 9120
9160 INPUT "UNIT WEIGHT (lb/gal)";ACON(I)
9170 PRINT: PRINT "NAME","UNITS","lb/gal"
9180 PRINT AUX$(I), UNIT$(I),
9190 IF UNIT$(I)="gal" THEN PRINT ACON(I);
9200 PRINT TAB(42);
9210 INPUT " CORRECT <Y or N>";Q$
9220 IF Q$="Y" OR Q$="y" THEN 9230 ELSE 9070
9230 NEXT I
9240 FOR I=1 TO 7
9250 AOWF(I)=0!
9260 NEXT I
9270 GOSUB 10500
9280 PRINT: PRINT: PRINT "INPUT % o.w.f."
9290 FOR I=1 TO 7
9300 IF AUX$(I)="        " THEN 9330
9310 LOCATE (2+I),30,1
9320 INPUT "",AOWF(I)
9330 NEXT I
9340 GOSUB 10500
9350 PRINT: PRINT: INPUT "CORRECT <Y or N> ";Q$
9360 IF Q$="Y" OR Q$="y" THEN 9380
9370 GOTO 9270
9380 SYS%=LASTREC
9390 GOSUB 7500
9400 RETURN
9410 END
```

```
9500 REM  EDIT SYSTEM
9510 PRINT "YOU HAVE INDICATED THAT THE NAMES OF DYES AND/OR AUXILIARY"
9520 PRINT "CHEMICALS USED TO DYE THIS SHADE HAVE CHANGED."
9530 PRINT
9540 PRINT "WILL *ALL* SHADES IN THIS STYLE FORMERLY DYED WITH THESE DYES AND"
9550 INPUT "AUXILIARIES ALSO HAVE THE SAME CHANGES <Y or N> ";Q$
9560 IF Q$="N" OR Q$="n" THEN 8300
9570 GOSUB 10600
9580 GFLAG=0
9590 LOCATE 18,1,1
9600 INPUT "DO WAVELENGTHS CHANGE ";Q$
9610 IF Q$="N" OR Q$="n" THEN 9710
9620 PRINT: PRINT "TYPE IN NEW WAVELENGTH AND 'RETURN' TO MAKE CHANGE"
9630 PRINT "OR, FOR NO CHANGE, JUST HIT 'RETURN'"
9640 GFLAG=1
9650 FOR I=1 TO N
9660 LOCATE 1,(9*I+15),1
9670 INPUT "",CHANGES$
9680 IF CHANGES$="" THEN 9700
9690 GAMMA(I)=VAL(CHANGES$)
9700 NEXT I
9710 LOCATE 18,1,1
9720 INPUT "DO DYESTUFF NAMES CHANGE ";Q$
9730 IF Q$="N" OR Q$="n" AND GFLAG<.5, THEN 9900
9740 PRINT: PRINT "TYPE IN NEW DYE NAMES AND K-VALUES AND 'RETURN' AFTER EACH"
9750 PRINT "OR, FOR NO CHANGE, JUST HIT 'RETURN'"
9760 FOR I=1 TO N
9770 LOCATE (2+I),2,1
9780 INPUT "",CHANGES$
9790 IF CHANGES$="" THEN 9820
9800 IF LEN(CHANGES$) >=5 THEN DYE$(I)=LEFT$(CHANGES$,5): GOTO 9830
9810 DYE$(I)=CHANGES$ + STRING$(5-LEN(CHANGES$)," ")
9820 IF GFLAG<.5 THEN 9890
9830 FOR J=1 TO N
9840 LOCATE (2+I),(8*J+14),1
9850 INPUT "",CHANGES$
9860 IF CHANGES$="" THEN 9880
9870 K(J,I)=VAL(CHANGES$)
9880 NEXT J
9890 NEXT I
9900 GOSUB 10600
9910 LOCATE 23,1,1
9920 INPUT "CORRECT <Y or N> ";Q$
9930 IF Q$="Y" OR Q$="y" THEN 9950
9940 GOTO 9570
9950 CLS: INPUT "DO AUXILIARY NAMES OR UNITS CHANGE <Y or N> ";Q$
9960 IF Q$="N" OR Q$="n" THEN 10240
9970 GOSUB 10700
9980 FOR I=1 TO 7
9990 LOCATE 21,1,1
10000 PRINT "TYPE IN NEW AUXILIARY NAME AND HIT 'RETURN' TO MAKE CHANGE"
```

```
10010 PRINT "OR, FOR NO CHANGE, HIT 'RETURN' ONLY"
10020 PRINT "OR, TO DELETE, TYPE 'D'"
10030 LOCATE (2+I),2,1
10040 INPUT "",CHANGES$
10050 IF CHANGES$="D" OR CHANGES$="d" THEN AUX$(I)="        ": UNIT$(I)="   ": ACON(I)=0!: AOWF(I)=0
!: GOTO 10230
10060 IF CHANGES$="" THEN 10090
10070 IF LEN(CHANGES$) >= 8 THEN AUX$(I)=LEFT$(CHANGES$,8): GOTO 10090
10080 AUX$(I)=CHANGES$ + STRING$(8-LEN(CHANGES$)," ")
10090 IF AUX$(I)="        " THEN 10230
10100 LOCATE 21,1,1
10110 PRINT "TYPE AUXILIARY UNITS: 'GAL' FOR GALLONS OR 'LBS' FOR POUNDS"
10120 LOCATE (2+I),21,1
10130 INPUT "",CHANGES$
10140 IF CHANGES$="" THEN 10160
10150 UNIT$(I)=CHANGES$
10160 IF LEFT$(UNIT$(I),1)="l" OR LEFT$(UNIT$(I),1)="L" THEN ACON(I)=0: GOTO 10230
10170 LOCATE 21,1,1: PRINT STRING$(80," "): LOCATE 21,1,1
10180 PRINT "TYPE UNIT WEIGHT (lb/gal)"
10190 LOCATE (2+I),32,1
10200 INPUT "",CHANGES$
10210 IF CHANGES$="" THEN 10230
10220 ACON(I)=VAL(CHANGES$)
10230 NEXT I
10240 GOSUB 10700
10250 LOCATE 23,1,1
10260 INPUT "CORRECT <Y or N> ";Q$
10270 IF Q$="Y" OR Q$="y" THEN 10290
10280 GOTO 9970
10290 INPUT "DO AUXILIARY CHEMICAL % o.w.f. VALUES CHANGE <Y or N> ";Q$
10300 IF Q$="N" OR Q$="n" THEN 10430
10310 REM  ENTRY POINT FOR AUXILIARY OWF CHANGES
10320 GOSUB 10500
10330 LOCATE 22,1,1
10340 PRINT "TYPE NEW % o.w.f. AND HIT 'RETURN' TO MAKE CHANGE"
10350 PRINT "OR, FOR NO CHANGE, HIT 'RETURN' ONLY"
10360 FOR I=1 TO 7
10370 IF AUX$(I)="        " THEN 10420
10380 LOCATE (2+I),30,1
10390 INPUT "",CHANGES$
10400 IF CHANGES$="" THEN 10420
10410 AOWF(I)=VAL(CHANGES$)
10420 NEXT I
10430 GOSUB 10500
10440 LOCATE 23,1,1
10450 INPUT "CORRECT <Y or N> ";Q$
10460 IF Q$="Y" OR Q$="y" THEN 10480
10470 GOTO 10320
10480 GOSUB 7500
10490 RETURN
10495 END
```

```
10500 REM  LIST AUXILIARY OWF
10510 CLS: PRINT "AUXILIARY","UNITS","% o.w.f.": PRINT
10520 FOR I=1 TO 7
10530 IF AUI$(I)="       " THEN 10550
10540 PRINT AUX$(I),UNIT$(I),: PRINT USING "##.##"; AOWF(I)
10550 NEXT I
10560 RETURN
10570 END
```

```
10600 REM  LIST DYES
10610 CLS
10620 PRINT TAB(1) "WAVELENGTH (nm)" TAB(23) GAMMA(1) TAB(32) GAMMA(2) TAB(41);
10630 PRINT GAMMA(3) TAB(50) GAMMA(4)
10640 PRINT
10650 FOR I=1 TO N
10660 PRINT TAB(2) DYE$(I) TAB(20);
10670 PRINT USING "#####.##"; K(1,I),K(2,I),K(3,I),K(4,I)
10680 NEXT I
10690 RETURN
10695 END
```

```
10700 REM  LIST AUXILIARIES
10710 CLS
10720 PRINT "AUXILIARY" TAB(20) "UNITS" TAB(30) "WT (gal/lb)": PRINT
10730 FOR I=1 TO 7
10740 IF AUX$(I)="       " THEN 10780
10750 PRINT TAB(2) AUX$(I) TAB(21) UNIT$(I) TAB(31);
10760 IF ACON(I)>.01, THEN PRINT ACON(I);
10770 PRINT " "
10780 NEXT I
10790 RETURN
10795 END
```

```
10800 REM  LIST ALL RECIPES
10810 CLS
10820 PRINT "WHERE DO YOU WANT THE RECIPES LISTED?"
10830 INPUT "TYPE '1' FOR SCREEN OR '2' FOR PRINTER ",OUTLIST%
10840 CLS
10850 I=1
10860 GET #2,I
10870 IF EOF(2) THEN 11090
10880 GOSUB 11300
10890 IF OUTLIST%=2 THEN LPRINT:LPRINT:LPRINT ELSE PRINT:PRINT:PRINT
10900 IF OUTLIST%=2 THEN LPRINT "STYLE" SPC(5) "SHADE  "; ELSE PRINT "STYLE" SPC(5) "SHADE  ";
10910 IF OUTLIST%=2 THEN LPRINT USING "\     \";DYE$(1);DYE$(2);DYE$(3);DYE$(4);
      ELSE PRINT USING "\     \";DYE$(1);DYE$(2);DYE$(3);DYE$(4);
10920 IF OUTLIST%=2 THEN LPRINT USING "\   \";AUX$(1);AUX$(2);AUX$(3);AUX$(4);AUX$(5);AUX$(6);AUX$(7)
      ELSE PRINT USING "\   \";AUX$(1);AUX$(2);AUX$(3);AUX$(4);AUX$(5);AUX$(6);AUX$(7)
10930 IF OUTLIST%=2 THEN LPRINT ELSE PRINT
10940 J=1
10950 GET #1,J
10960 IF EOF(1) THEN 11070
10970 SYSNO%=CVI(C$)
10980 IF SYSNO%<>SYS% THEN 11050
10990 STY%=CVI(A$)
11000 SHD=CVS(B$)
11010 GOSUB 11200
11020 IF OUTLIST%=2 THEN LPRINT LEFT$(STYLE$(STY%),8) TAB(10) SHD; ELSE PRINT LEFT$(STYLE$(STY%),8)
      TAB(10) SHD;
11030 IF OUTLIST%=2 THEN LPRINT USING "##.####"; DOWF(1);DOWF(2);DOWF(3);DOWF(4); ELSE PRINT USING
      "##.####"; DOWF(1);DOWF(2);DOWF(3);DOWF(4);
11040 IF OUTLIST%=2 THEN LPRINT USING "##.##";AOWF(1);AOWF(2);AOWF(3);AOWF(4);AOWF(5);AOWF(6);
      AOWF(7) ELSE PRINT USING "##.##"; AOWF(1);AOWF(2);AOWF(3);AOWF(4);AOWF(5);AOWF(6);AOWF(7)
11050 J=J+1
11060 GOTO 10950
11070 I=I+1
11080 GOTO 10860
11090 INPUT "HIT 'RETURN' TO CONTINUE",ANY$
11100 RETURN
11110 END
```

```
11200 REM  CONVERT RECIPE RECORD
11210 DOWF(1)=CVS(E$)
11220 DOWF(2)=CVS(G$)
11230 DOWF(3)=CVS(I$)
11240 DOWF(4)=CVS(K$)
11250 SYSNO%=CVI(C$)
11260 RETURN
11270 END
```

```
11300 REM  CONVERT SYSTEM RECORD - PART 1
11310 AOWF(1)=CVS(D$)
11320 AOWF(2)=CVS(R$)
11330 AOWF(3)=CVS(T$)
11340 AOWF(4)=CVS(V$)
11350 AOWF(5)=CVS(X$)
11360 AOWF(6)=CVS(Z$)
11370 AOWF(7)=CVS(BB$)
11380 SYS%=CVI(CC$)
11390 SYSTY%=CVI(X3$)
11400 N=CVI(NN$)
11410 ACON(1)=CVS(N$)
11420 ACON(2)=CVS(P$)
11430 ACON(3)=CVS(S$)
11440 ACON(4)=CVS(U$)
11450 ACON(5)=CVS(W$)
11460 ACON(6)=CVS(Y$)
11470 ACON(7)=CVS(AA$)
11480 FOR IC=1 TO 7
11490 IF ACON(IC)>.01, THEN UNIT$(IC)="gal": GOTO 11510
11500 UNIT$(IC)="lbs"
11510 NEXT IC
11520 DYE$(1)=A3$
11530 DYE$(2)=B3$
11540 DYE$(3)=C3$
11550 DYE$(4)=D3$
11560 AUX$(1)=E3$
11570 AUX$(2)=F3$
11580 AUX$(3)=G3$
11590 AUX$(4)=H3$
11600 AUX$(5)=I3$
11610 AUX$(6)=J3$
11620 AUX$(7)=K3$
11630 RETURN
11640 END
```

```
11700 REM  CONVERT SYSTEM RECORD - PART 2
11710 GAMMA(1) = CVI(A1$)
11720 GAMMA(2) = CVI(B1$)
11730 GAMMA(3) = CVI(C1$)
11740 GAMMA(4) = CVI(D1$)
11750 K(1,1) = CVS(A2$)
11760 K(1,2) = CVS(B2$)
11770 K(1,3) = CVS(C2$)
11780 K(1,4) = CVS(D2$)
11790 K(2,1) = CVS(E2$)
11800 K(2,2) = CVS(F2$)
11810 K(2,3) = CVS(G2$)
11820 K(2,4) = CVS(H2$)
11830 K(3,1) = CVS(I2$)
11840 K(3,2) = CVS(J2$)
11850 K(3,3) = CVS(K2$)
11860 K(3,4) = CVS(L2$)
11870 K(4,1) = CVS(M2$)
11880 K(4,2) = CVS(N2$)
11890 K(4,3) = CVS(O2$)
11900 K(4,4) = CVS(P2$)
11910 RETURN
11920 END
```

```
12000 REM  END
12010 CLOSE
12020 SYSTEM
12030 PRINT "RUN ERROR - PROGRAM ABORT - FILES SAVED"
12040 CLOSE
12050 SYSTEM
```

# APPENDIX B

# QUALITY ASSURANCE PROCEDURES

Because this project represented the first development of data on the environmental benefits of dyebath reuse, strict quality assurance procedures were used to ensure that valid environmental data were collected. Quality assurance procedures were applied to all areas of work during this project. Specific areas dealing with environmental data collection included flow and volume measurements, wastewater sampling, pollutant analysis, and data calculations.

## Flow/Volume Measurements

The pilot-scale dyebeck was calibrated in 5.0-liter increments to yield a depth-of-water versus volume calibration. Depth measurements were made and verified during the dyeings to the nearest 1/4-inch, or 2.5 liters. This yields an accuracy of about 3 percent.

The full-scale dyebecks were calibrated using a 4-inch totalizing magnetic flow meter to yield a depth-of-water versus volume calibration. Depth measurements were taken and verified during the full-scale dyeings. The accuracy of these beck volume measurements is about 5 percent. Pumped dyebath volume measurements were made directly with the totalizing magnetic flow meter. The calibration of the flow meter was factory-tested and showed an accuracy of better than 1 percent for flow rates from 130 to 1,280 gpm. Flow rates observed during pumping ranged from 200 to 600 gpm.

## Wastewater Sampling

The procedures used to assure quality in sampling, bottling, preserving, and shipping of wastewater samples are described in Section IX of this report.

## Pollutant Analysis

Procedures used to assure the quality of field pH and temperature measurements are described in Section IX of this report. The calibration of the pH meter was checked daily.

Procedures used to assure the quality of laboratory analyses included the use of duplicate analyses, spikes, and check samples. Duplicate analyses provide a measure of the precision of the analytical technique. Spikes and check samples provide a measure of accuracy. The quality control data generated for this project are reported on the following pages.

### Calculations and Reports

All final project calculations and reports were subjected to a quality control procedure consisting of document checking, backchecking, rechecking, and review. The procedure is intended to verify the accuracy of data and calculations, and the accuracy of the technical content and supporting documentation for reports.

# DUPLICATE ANALYSES

| BOD | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 17-3 | 1,670 | 1,580 |
| | 19-2 | 1,670 | 1,440 |
| | 19-5 | 2,140 | 1,970 |
| | 20-1 | 770 | 540 |
| | 21-4 | 1,940 | 1,870 |

| COD | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 17-5 | 5,790 | 5,830 |
| | 23-10 | 7,260 | 7,260 |

| TDS | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 18-5 | 1,720 | 1,730 |
| | 19-4 | 1,720 | 1,740 |
| | 19-5 | 1,800 | 1,710 |
| | 23-1 | 1,600 | 1,620 |
| | 23-2 | 2,150 | 2,110 |
| | 25-2A | 1,760 | 1,690 |
| | 25-3C | 380 | 380 |
| | 26-4A | 1,770 | 1,980 |
| | 26-5C | 470 | 580 |
| | 26-8C | 200 | 220 |
| | 26-10A | 2,290 | 2,710 |
| | Plant Water | 150 | 140 |

| TSS | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 18-5 | 112 | 100 |
| | 19-4 | 45 | 34 |
| | 19-5 | 137 | 111 |
| | 23-1 | 5 | 10 |
| | 23-2 | 11 | 9 |
| | 25-2A | 24 | 29 |
| | 25-3C | 6 | 5 |
| | 26-1A | 6 | 7 |
| | 26-4A | 15 | 21 |
| | 26-5C | 4 | 11 |
| | 26-8C | 26 | 17 |
| | 26-10A | 88 | 67 |
| | Plant Water | 0 | 2 |

| Total P | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 17-5 | 119 | 120 |
| | 19-4 | 85 | 88 |
| | 21-1 | 71 | 65 |
| | 22-1 | 60 | 71 |
| | 23-6 | 45 | 45 |

## DUPLICATE ANALYSES (continued)

| Alkalinity | Sample No. | Results (mg/l) | |
|---|---|---|---|
| | 17-5 | 139 | 139 |
| | 19-5 | 133 | 133 |

| Phenols | Sample No. | Results (ug/l) | |
|---|---|---|---|
| | 18-5 | 56 | 61 |
| | 19-1 | 73 | 73 |
| | 19-2 | 81 | 82 |
| | 19-4 | 122 | 157 |
| | 19-5 | 87 | 97 |
| | 24-1B | 87 | 54 |
| | 24-1C | 57 | 57 |
| | 24-2C | 189 | 93 |
| | 25-4C | <6 | 10 |
| | 26-5A | <6 | <6 |

## SPIKE RECOVERY

| | Sample No. | Concentration | Spike | Recovery |
|---|---|---|---|---|
| Alkalinity | 21-3 | 153 mg/l | 99.6 mg/l | 96.3% |
| Alkalinity | 23-2 | 135 mg/l | 99.6 mg/l | 94.3% |

## CHECK SAMPLES

| | Should be | Recovery |
|---|---|---|
| COD | 233 mg/l | 97.8% |
| BOD | 220 mg/l | 96.8% |